PB89-181945

USEPA (United States Environmental Protection Agency) Method Study 38, SW-846, Method 3010 Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by Flame Atomic Absorption Spectroscopy

Bionetics Corp., Cincinnati, OH

Prepared for

Environmental Monitoring Systems Lab.
Cincinnati, OH

Apr 89

PB89-181945

EPA/600/4-89/011
April 1989

USEPA METHOD STUDY 38

SW-846 METHOD 3010

ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS FOR ANALYSIS BY FLAME ATOMIC ABSORPTION SPECTROSCOPY

by

K.W. Edgell and D.M. Wilbers
The Bionetics Corporation
16 Triangle Park Drive
Cincinnati, Ohio 45246

Contract No. 68-03-3254

Project Officer

Edward L. Berg
Quality Assurance Research Division
Environmental Monitoring Systems Laboratory
Cincinnati, Ohio 45268

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
US ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

REPRODUCED BY
U.S. DEPARTMENT OF COMMERCE
NATIONAL TECHNICAL INFORMATION SERVICE
SPRINGFIELD, VA. 22161

TECHNICAL REPORT DATA

(Please read Instructions on the reverse before completing)

1. REPORT NO.
EPA/600/4-89/011

2.
846

3. RECIPIENT'S ACCESSION NO.
PB89 181945/AS

4. TITLE AND SUBTITLE
U.S. EPA Method Study #38 - SW/Method 3010, Acid Digestion of Aqueous Samples and Extracts for Trace Metals by Flame Atomic Absorption Spectroscopy

5. REPORT DATE
April 1989

6. PERFORMING ORGANIZATION CODE

7. AUTHOR(S)
Kenneth W. Edgell and D. M. Wilbers

8. PERFORMING ORGANIZATION REPORT NO.

9. PERFORMING ORGANIZATION NAME AND ADDRESS
Environmental Monitoring Systems Laboratory
Quality Assurance Research Division, USEPA
26 W. Martin Luther King Drive
Cincinnati, OH 45268

10. PROGRAM ELEMENT NO.
CBSD1A

11. CONTRACT/GRANT NO.
68-03-3254

12. SPONSORING AGENCY NAME AND ADDRESS
Environmental Monitoring Systems Laboratory
Quality Assurance Research Division, USEPA
26 W. Martin Luther King Drive
Cincinnati, OH 45268

13. TYPE OF REPORT AND PERIOD COVERED
Final Report 9-86 to 2-88

14. SPONSORING AGENCY CODE
EPA 600/06

15. SUPPLEMENTARY NOTES

16. ABSTRACT

An interlaboratory collaborative study was conducted on SW-846 Method 3010, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by Flame Atomic Absorption Spectroscopy", to determine the mean recovery and precision for analyses of 21 trace metals in surface and wastewaters.

The study design was based upon Youden's non-replicate plan for collaborative tests of analytical methods. Four water matrices were spiked with 21 trace metals at six concentration levels, as three Youden pairs. Nine participating laboratories analyzed the four sample types using Method 3010. The primary objective was to use the USEPA computer program entitled "Interlaboratory Method Validation Study" (IMVS) to measure recovery and precision for the 21 trace metals and compare the performance of the method across water types. Minimum detection limits (MDLs) were also compared with MDLs reported in the SW-846 methods manual. Comparison between samples, at the lowest metals concentration specified in the SW-846 and another sample at 50% of those values, verified that the values specified in SW-846 are valid.

The studies were conducted by the Bionetics Corporation under the direction of the Quality Assurance Research Division, Environmental Monitoring Systems Laboratory, Cincinnati, OH under Contract No. 68-03-3254. Analytical work was completed as of November 30, 1987. The reports cover a period from September 10, 1986 to February 28, 1988.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group

18. DISTRIBUTION STATEMENT
Release to Public

19. SECURITY CLASS *(This Report)*
Unclassified

20. SECURITY CLASS *(This page)*
Unclassified

21. NO. OF PAGES
178

22. PRICE

DISCLAIMER

The information contained in this document has been funded wholly by the United States Environmental Protection Agency to The Bionetics Corporation, Cincinnati, Ohio under Contract No. 68-03-3254. It has been subjected to Agency peer and administrative review and has been approved for publication as an EPA document. Mention of trade names or commercial products does not constitute endorsement or recommendation for use by the United States Environmental Protection Agency.

FOREWORD

Environmental measurements are required to determine the quality of ambient waters and the character of wastes and wastewater effluents. The Environmental Monitoring Systems Laboratory (EMSL)-Cincinnati, conducts research to:

Develop and evaluate techniques to measure the presence and concentration of physical and chemical pollutants in water, wastewater, bottom sediments, and solid waste.

Investigate methods for the concentration, recovery, and identification of viruses, bacteria, and other microorganisms in water.

Determine the response of aquatic organisms to water quality.

Support an Agency-wide quality assurance program to assure standardization and quality control of systems for monitoring waste and wastewater.

This publication of EMSL-Cincinnati, entitled EPA Method Study 38, SW-846 Method 3010, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by Flame Atomic Absorption Spectroscopy, reports the results of EPA's interlaboratory method validation study for the analyses of 21 trace elements in reagent water, surface water and two wastewaters.

Federal agencies, states, municipalities, universities, private laboratories, and industry should find this evaluative study of assistance in monitoring and controlling pollution in the environment.

Thomas A. Clark, Director
EMSL - Cincinnati
February 1988

ABSTRACT

An interlaboratory method validation study was conducted on SW-846 Method 3010, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by Flame Atomic Absorption Spectroscopy", to determine the mean recovery and precision for analyses of 21 trace metals in surface and wastewaters. SW-846 Method 3010 includes instructions for quality control, sample preparation and analysis of samples by flame atomic absorption spectroscopy (FLAA).

The study design was based upon Youden's non-replicate plan for collaborative tests of analytical methods (1). Four water matrices were spiked with 21 trace metals at six concentration levels, as three Youden pairs. Nine laboratories digested and refluxed the spiked water matrices with concentrated nitric acid, brought them to volume with dilute hydrochloric acid, and analyzed each for 21 trace metals by FLAA. The primary objective of the study was to analyze the results using USEPA computer programs entitled "Interlaboratory Method Validation Study" (IMVS) (2) which produced measures of recovery and precision for the 21 trace metals and compared the performance of the method between water types.

Two additional studies were also conducted using Method 3010. One study compared minimum detection limits (MDLs) reported in the SW-846 method manual to MDLs determined using Method 3010. Another study was conducted to verify that the lowest concentration levels of the optimum range given in the SW-846 method manual are valid. Comparison between the samples at the lowest concentration level specified in SW-846 and another sample at 50% that value were performed for mean recovery, overall precision, and single-analyst precision.

The studies were conducted by The Bionetics Corporation under the direction of the Quality Assurance Research Division, Environmental

Monitoring Systems Laboratory, Cincinnati, OH (EMSL-Cincinnati) under Contract No. 68-03-3254. Analytical work was completed as of November 30, 1987. The report covers a period from September 10, 1986 to February 28, 1988.

TABLE OF CONTENTS

Foreword iii
Abstract iv
Tables viii
Figures xi
Acknowledgments xii
1. Introduction 1
2. Conclusions. 4
3. Recommendations. 13
4. Description of the Study 14
    Method Summary 14
    Study Design 14
    Verification Analyses 15
    Selection of Test Waters 15
    Selection of Participating Laboratories 16
5. Treatment of Data 18
    Rejection of Outliers 18
    Youden's Laboratory Ranking Procedure 19
    Tests for Individual Outliers 20
    Statistical Data Summaries 20
    Statement of Method Recovery 23
    Statement of Method Precision 24
    Comparison of Recovery and Precision Across Water Types 26
6. Results and Discussion 32
    Rejection of Outliers 32
    Mean Recovery 34
    Precision 36
    Effects of Water Type 39
    Method Detection Limits (MDLs) 49
    Evaluation of Lowest Concentration Levels 49

TABLE OF CONTENTS (Continued)

References 55
Appendices
A. Laboratory Instructions - SW-846 Method 3010 57
B. Raw Data 73
C. Statistical Summaries. 116
D. Effect of Water Type 138
E. Method Detection Limit Raw Data. 160

TABLES

NUMBER — Page

1. Weighted Linear Regression Equations for Mean Recovery and Precision 5
2. ICP Results of Background Screening Analyses of the Three Matrix Waters Used in the Method 3010 Study 17
3. Number of Rejected Data Points by Element, By Water Type, and in Total. 33
4. Number of Rejected Data Points by Laboratory and Outlier Test . . 35
5. Estimated Mean Recovery Calculated from Recovery Regression Equations, for SW-846 Method 3010, as Percent of True Value . 37
6. Estimated Overall Precision by Element and Water Type, as %RSD, Calculated from the Precision Regression Equations in Table 1 40
7. Estimated Single-Analyst Precision by Element and Water Type, as %RSD-SR, Calculated from the Precision Regression Equations in Table 1 41
8. Effect of Water Type 43
9. Comparison of Measured Method Detection Limits (MDLs) Versus SW-846 Method Detection Limits 50
10. Comparison of Recovery in Reagent Water of Samples 5 & 6 Versus Samples 7 & 8, as Percent of True Value 51
11. Comparison of Overall Precision in Reagent Water, as %RSD, of Samples 5 & 6 Versus Samples 7 & 8. 53
12. Comparison of Single-Analyst Precision in Reagent Water, as %RSD-SR, of Samples 5 & 6 Versus Samples 7 & 8. 54
13. Reference Values for QC Samples 60
14. Raw Data for Aluminum 74
15. Raw Data for Antimony 76
16. Raw Data for Barium 78
17. Raw Data for Beryllium 80
18. Raw Data for Cadmium 82

TABLES (CONTINUED)

NUMBER PAGE

19. Raw Data for Calcium 84
20. Raw Data for Chromium 86
21. Raw Data for Cobalt 88
22. Raw Data for Copper 90
23. Raw Data for Iron 92
24. Raw Data for Lead 94
25. Raw Data for Magnesium 96
26. Raw Data for Manganese 98
27. Raw Data for Molybdenum 100
28. Raw Data for Nickel 102
29. Raw Data for Potassium 104
30. Raw Data for Silver 106
31. Raw Data for Sodium 108
32. Raw Data for Thallium 110
33. Raw Data for Vanadium 112
34. Raw Data for Zinc 114
35. Statistical Summary for Aluminum 117
36. Statistical Summary for Antimony 118
37. Statistical Summary for Barium 119
38. Statistical Summary for Beryllium 120
39. Statistical Summary for Cadmium 121
40. Statistical Summary for Calcium 122
41. Statistical Summary for Chromium 123
42. Statistical Summary for Cobalt 124
43. Statistical Summary for Copper 125
44. Statistical Summary for Iron 126
45. Statistical Summary for Lead 127
46. Statistical Summary for Magnesium 128
47. Statistical Summary for Manganese 129
48. Statistical Summary for Molybdenum 130
49. Statistical Summary for Nickel 131
50. Statistical Summary for Potassium 132

TABLES (CONTINUED)

NUMBER PAGE

51. Statistical Summary for Silver 133
52. Statistical Summary for Sodium 134
53. Statistical Summary for Thallium 135
54. Statistical Summary for Vanadium 136
55. Statistical Summary for Zinc 137
56. Effect of Water Type for Aluminum 139
57. Effect of Water Type for Antimony 140
58. Effect of Water Type for Barium 141
59. Effect of Water Type for Beryllium 142
60. Effect of Water Type for Cadmium 143
61. Effect of Water Type for Calcium 144
62. Effect of Water Type for Chromium 145
63. Effect of Water Type for Cobalt 146
64. Effect of Water Type for Copper 147
65. Effect of Water Type for Iron 148
66. Effect of Water Type for Lead 149
67. Effect of Water Type for Magnesium 150
68. Effect of Water Type for Manganese 151
69. Effect of Water Type for Molybdenum 152
70. Effect of Water Type for Nickel 153
71. Effect of Water Type for Potassium 154
72. Effect of Water Type for Silver 155
73. Effect of Water Type for Sodium 156
74. Effect of Water Type for Thallium 157
75. Effect of Water Type for Vanadium 158
76. Effect of Water Type for Zinc 159
77. Measured MDLs in Reagent Water by Laboratory 161
78. Measured MDLs in Ohio River Water by Laboratory 162
79. Measured MDLs in Municipal Secondary Effluent by Laboratory . . 163
80. Measured MDLs in Electroplating Effluent by Laboratory 164

FIGURES

NUMBER **PAGE**

1. Recovery of Silver as Percent of Spiked Concentration 38

ACKNOWLEDGEMENTS

The author gratefully acknowledges the direction and cooperation provided by the staffs of the Quality Assurance Research Division and Chemistry Research Division, EMSL-Cincinnati and the technical staff at The Bionetics Corporation, particularly Dawn Wilbers, the primary analyst, and Carol Pfirman and Elinor Hazel for many hours of patient typing. Special thanks are expressed to Mr. John Ortman for data processing, to Mr.Theodore Martin for his analytical assistance, to the analysts of the nine laboratories participating in this study and to Mr. Edward Berg, Project Officer, for his leadership and direction throughout this study.

SECTION 1

INTRODUCTION

The Hazardous Waste Management Facility Permit Regulations were promulgated in July, 1982 (40 CFR 265) and provide performance standards for the monitoring of ground waters, wastewaters and solid wastes at hazardous waste sites. To facilitate these standards, certain analytical methodology are required to assess the degree of contamination at and around the area of the site. The Manual: Test Methods for Evaluating Solid Waste, Physical and Chemical Methods, (SW-846), November 1986, Third Edition, provides a unified, up-to-date source of information on sampling and analyses related to compliance with Resource Conservation and Recovery Act (RCRA) regulations. The success of these pollution control activities, particularly when legal action is involved, depends upon the reliability of the data provided by the laboratories. Therefore, it is important to evaluate the methodology through interlaboratory method validation studies.

The Environmental Monitoring Systems Laboratory, Cincinnati, OH (EMSL-Cincinnati) develops or selects analytical methods and provides quality assurance (QA) support for Agency programs involving water and wastewater regulations. In EMSL-Cincinnati the responsibility for providing support is assigned to the Quality Assurance Research Division (QARD). Its program is designed to provide the QA support needed to establish the reliability and legal defensibility of water and wastewater data collected by the Agency, the state regulating authorities, the private sector, and commercial laboratories performing compliance analyses. One of QARD's QA activities is to conduct interlaboratory method validation studies to evaluate analytical methods selected for the Agency's operating programs such as the Office of Solid Waste.

This report describes an interlaboratory method validation study for SW-846 Method 3010, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by Flame Atomic Absorption Spectroscopy". The elements: aluminum, antimony, barium, beryllium, calcium, cadmium, chromium, cobalt, copper, iron, lead, magnesium, manganese, molybdenum, nickel, potassium, silver, sodium, thallium, vanadium, and zinc were analyzed using Method 3010.

The primary objective of the study was to characterize the behavior of Method 3010 in terms of recovery, overall and single-analyst precision, and the effect of water type on recovery and precision. The study was conducted with the cooperation of nine participating laboratories under the direction of the Quality Assurance Research Division, EMSL-Cincinnati. The Bionetics Corporation, as primary contractor to QARD, was responsible for the collection and characterization of the water matrices, preparation of analyte spiking solutions, preparation of user instructions and report forms, distribution of samples and screening the returned data for gross errors. The raw data were evaluated statistically by the QARD using a series of computer programs entitled, "Interlaboratory Method Validation Studies" (IMVS). Upon review of the draft report by EMSL-Cincinnati, The Bionetics Corporation prepared the final report.

A second objective of the study was to compare the minimum detection limits (MDLs) reported in the SW-846 methods manual to the MDLs determined for the 21 elements using Method 3010. The MDL for each element was obtained by measuring the noise level response for seven reagent water digestates with no detectable background. The mean and standard deviation of the noise level responses was then calculated. Then each of the four water matrices was digested and used to prepare a 5 point calibration curve using the Method of Standard Additions. The MDLs were calculated using the following relationship:

$$MDL = 3(SD)/m$$

where, m = (y-i)/x = slope of the calibration curve
x = concentration (ug/mL)
y = signal at concentration x
i = intercept of the calibration curve
SD = standard deviation of the noise level responses

A third study objective was to verify that the lowest concentration levels of the optimum range specified in the SW-846 method manual are valid. Comparison between the samples at the lowest concentration level and another sample at 50% that value was performed for mean recovery, overall precision, and single-analyst precision.

SECTION 2

CONCLUSIONS

The primary objective of this study was to characterize the performance of Method 3010 in terms of recovery, overall precision, single-analyst precision, and the effect of water types on recovery and precision. Through the IMVS computer programs, statistical analyses of 4536 analytical values provided estimates of recovery and precision expressed as regression equations which are presented in Table 1. These equations may be used to predict the recovery and precision of the 21 elements over the range tested.

The IMVS programs rejected 995 data points (21.9%) of the 4536 data points submitted. The percentage of rejected data did not vary significantly among the water types, ranging from 20% for reagent water to 28% for Ohio River Water. Thallium had the fewest rejected data with 15 while beryllium had the most with 78. The number of rejected data by laboratory ranged from 27 for Lab 7 to 234 for Lab 6. The rejected data were not evenly distributed among the laboratories. Labs 3, 4, 6 and 9 accounted for 624 or 62.7% of the 995 total rejected data.

Recovery estimates for the 21 elements were calculated at the midpoint concentration of the range studied using the regression equations in Table 1. The average percent recovery of the elements across all water types was excellent ranging from 94% to 109% with two exceptions, silver (28%) and barium (74%).

Silver recoveries were concentration dependent. The silver concentration range used in this study was 0.10 to 4.0 ug/mL as recommended in the SW-846 method manual. In reagent water the percent recoveries of the low sample concentration pair, 0.10 to 4.0 ug/mL, were 97.8% and 95.6% respectively. However, as the concentration levels increased, the percent recovery

TABLE 1 STUDY 38, SW 846 METHOD 3010, TRACE METALS, AA FLAME

WEIGHTED LINEAR REGRESSION EQUATIONS FOR MEAN RECOVERY AND PRECISION (IN UG/ML)

WATER TYPE	ALUMINUM, AL	BERYLLIUM, BE	CALCIUM, CA	CADMIUM, CD
APPLICABLE CONC. RANGE	(5.00 - 50.00)	(0.04 - 2.00)	(18.00 - 120.00)	(0.04 - 2.00)
REAGENT WATER				
SINGLE-ANALYST PRECISION	SR = 0.054$\bar{X}$ + 0.17	SR = 0.019$\bar{X}$ + 0.01	SR = 0.097$\bar{X}$ - 0.87	SR = 0.017$\bar{X}$ + 0.00
OVERALL PRECISION	S = 0.086$\bar{X}$ - 0.02	S = 0.118$\bar{X}$ + 0.00	S = 0.117$\bar{X}$ - 0.02	S = 0.029$\bar{X}$ + 0.00
MEAN RECOVERY	$\bar{X}$ = 1.026C - 0.29	$\bar{X}$ = 0.913C + 0.00	$\bar{X}$ = 1.001C + 2.12	$\bar{X}$ = 0.993C + 0.00
WATER 1				
SINGLE-ANALYST PRECISION	SR = 0.020$\bar{X}$ + 0.09	SR = 0.020$\bar{X}$ + 0.00	SR = 0.105$\bar{X}$ - 0.71	SR = 0.016$\bar{X}$ + 0.00
OVERALL PRECISION	S = 0.071$\bar{X}$ - 0.07	S = 0.054$\bar{X}$ - 0.00	S = 0.200$\bar{X}$ - 2.41	S = 0.034$\bar{X}$ + 0.00
MEAN RECOVERY	$\bar{X}$ = 0.947C - 0.25	$\bar{X}$ = 0.951C + 0.00	$\bar{X}$ = 0.938C - 1.36	$\bar{X}$ = 0.989C - 0.00
WATER 2				
SINGLE-ANALYST PRECISION	SR = 0.021$\bar{X}$ + 0.08	SR = 0.032$\bar{X}$ + 0.00	SR = 0.018$\bar{X}$ + 2.46	SR = 0.022$\bar{X}$ + 0.00
OVERALL PRECISION	S = 0.026$\bar{X}$ + 0.37	S = 0.048$\bar{X}$ + 0.00	S = 0.122$\bar{X}$ + 1.29	S = 0.042$\bar{X}$ + 0.01
MEAN RECOVERY	$\bar{X}$ = 0.993C - 0.32	$\bar{X}$ = 0.966C + 0.00	$\bar{X}$ = 1.051C + 0.97	$\bar{X}$ = 0.988C + 0.00
WATER 3				
SINGLE-ANALYST PRECISION	SR = 0.024$\bar{X}$ - 0.02	SR = 0.020$\bar{X}$ + 0.00	SR = 0.016$\bar{X}$ + 2.67	SR = 0.019$\bar{X}$ + 0.00
OVERALL PRECISION	S = 0.036$\bar{X}$ + 0.06	S = 0.038$\bar{X}$ + 0.00	S = 0.167$\bar{X}$ - 0.95	S = 0.029$\bar{X}$ + 0.01
MEAN RECOVERY	$\bar{X}$ = 1.019C - 0.21	$\bar{X}$ = 0.967C + 0.00	$\bar{X}$ = 0.898C + 2.28	$\bar{X}$ = 1.001C + 0.00

$\bar{X}$ = MEAN RECOVERY
C = TRUE VALUE FOR THE CONCENTRATION

WATER 1 - OHIO RIVER WATER
WATER 2 - MUNICIPAL SECONDARY EFFLUENT
WATER 3 - ELECTROPLATING EFFLUENT

TABLE 1 (cont.) STUDY 38, SW 846 METHOD 3010, TRACE METALS, AA FLAME

WEIGHTED LINEAR REGRESSION EQUATIONS FOR MEAN RECOVERY AND PRECISION (IN UG/ML)

WATER TYPE	COBALT, CO	CHROMIUM, CR	COPPER , CU	IRON, FE
APPLICABLE CONC. RANGE	(2.50 - 15.00)	(0.50 - 10.00)	(0.20 - 5.00)	(0.30 - 5.00)
REAGENT WATER				
SINGLE-ANALYST PRECISION	SR = 0.032X - 0.05	SR = 0.019X + 0.06	SR = 0.012X + 0.01	SR = 0.010X + 0.05
OVERALL PRECISION	S = 0.066X - 0.07	S = 0.090X + 0.01	S = 0.030X + 0.01	S = 0.051X + 0.03
MEAN RECOVERY	X = 0.985C + 0.01	X = 1.082C - 0.03	X = 0.997C + 0.00	X = 0.955C + 0.01
WATER 1				
SINGLE-ANALYST PRECISION	SR = 0.015X + 0.06	SR = 0.030X - 0.00	SR = 0.024X - 0.00	SR = 0.031X + 0.06
OVERALL PRECISION	S = 0.086X + 0.00	S = 0.079X + 0.03	S = 0.026X + 0.01	S = 0.058X + 0.10
MEAN RECOVERY	X = 0.992C - 0.09	X = 1.093C + 0.02	X = 0.988C + 0.00	X = 0.962C - 0.12
WATER 2				
SINGLE-ANALYST PRECISION	SR = 0.018X + 0.21	SR = 0.027X + 0.03	SR = 0.010X + 0.00	SR =-0.002X + 0.08
OVERALL PRECISION	S = 0.056X + 0.20	S = 0.071X + 0.03	S = 0.022X + 0.01	S = 0.027X + 0.10
MEAN RECOVERY	X = 0.961C + 0.39	X = 1.116C - 0.06	X = 0.993C + 0.01	X = 0.985C - 0.05
WATER 3				
SINGLE-ANALYST PRECISION	SR = 0.031X - 0.04	SR = 0.034X + 0.00	SR = 0.016X - 0.00	SR = 0.026X + 0.04
OVERALL PRECISION	S = 0.044X + 0.08	S = 0.088X + 0.07	S = 0.036X + 0.01	S = 0.075X + 0.06
MEAN RECOVERY	X = 0.990C - 0.05	X = 1.077C - 0.02	X = 0.997C + 0.00	X = 1.065C - 0.15

X = MEAN RECOVERY
C = TRUE VALUE FOR THE CONCENTRATION

WATER 1 - OHIO RIVER WATER
WATER 2 - MUNICIPAL SECONDARY EFFLUENT
WATER 3 - ELECTROPLATING EFFLUENT

TABLE 1 (cont.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

WEIGHTED LINEAR REGRESSION EQUATIONS FOR MEAN RECOVERY AND PRECISION (IN UG/ML)

WATER TYPE	MAGNESIUM, MG	MANGANESE, MN	MOLYBDENUM, MO	SODIUM, NA
APPLICABLE CONC. RANGE	(5.00 - 40.00)	(0.10 - 3.00)	(1.00 - 40.00)	(96.00 - 600.00)
REAGENT WATER				
SINGLE-ANALYST PRECISION	SR = 0.022X - 0.03	SR = 0.020X + 0.01	SR = 0.064X + 0.25	SR = 0.038X - 1.82
OVERALL PRECISION	S = 0.058X - 0.24	S = 0.030X + 0.01	S = 0.107X + 0.22	S = 0.068X - 0.42
MEAN RECOVERY	X = 1.027C - 0.02	X = 1.019C + 0.01	X = 1.031C - 0.012	X = 1.025C - 0.17
WATER 1				
SINGLE-ANALYST PRECISION	SR = 0.009X + 0.22	SR = 0.011X + 0.00	SR = 0.058X - 0.01	SR = 0.025X - 0.80
OVERALL PRECISION	S = 0.020X + 0.35	S = 0.020X + 0.02	S = 0.111X - 0.05	S = 0.038X + 0.37
MEAN RECOVERY	X = 1.018C - 0.10	X = 1.008C - 0.01	X = 1.011C + 0.019	X = 0.986C + 2.44
WATER 2				
SINGLE-ANALYST PRECISION	SR = 0.072X - 0.10	SR = 0.006X + 0.01	SR = 0.049X + 0.02	SR = 0.066X - 2.56
OVERALL PRECISION	S = 0.059X + 0.07	S = 0.027X + 0.01	S = 0.143X - 0.07	S = 0.066X + 2.03
MEAN RECOVERY	X = 1.021C - 0.20	X = 0.987C - 0.00	X = 1.051C + 0.107	X = 0.991C - 1.80
WATER 3				
SINGLE-ANALYST PRECISION	SR = 0.014X + 0.08	SR = 0.020X + 0.00	SR = 0.043X + 0.08	SR = 0.050X + 0.14
OVERALL PRECISION	S = 0.034X - 0.00	S = 0.048X + 0.00	S = 0.123X - 0.02	S = 0.033X +11.92
MEAN RECOVERY	X = 1.011C - 0.03	X = 1.024C + 0.00	X = 1.002C + 0.043	X = 0.988C - 8.91

X = MEAN RECOVERY
C = TRUE VALUE FOR THE CONCENTRATION

WATER 1 - OHIO RIVER WATER
WATER 2 - MUNICIPAL SECONDARY EFFLUENT
WATER 3 - ELECTROPLATING EFFLUENT

TABLE 1 (cont.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

WEIGHTED LINEAR REGRESSION EQUATIONS FOR MEAN RECOVERY AND PRECISION (IN UG/ML)

WATER TYPE	NICKEL, NI	LEAD, PB	ANTIMONY, SB	THALLIUM, TI
APPLICABLE CONC. RANGE	(0.30 - 5.00)	(0.80 - 20.00)	(1.20 - 40.00)	(1.00 - 20.00)
REAGENT WATER				
SINGLE-ANALYST PRECISION	SR = 0.004X + 0.02	SR = 0.018X + 0.03	SR = 0.023X + 0.19	SR = 0.009X + 0.07
OVERALL PRECISION	S = 0.029X + 0.03	S = 0.019X + 0.09	S = 0.044X + 0.20	S = 0.021X + 0.05
MEAN RECOVERY	X = 0.991C - 0.01	X = 1.031C - 0.02	X = 1.057C - 0.25	X = 1.039C - 0.03
WATER 1				
SINGLE-ANALYST PRECISION	SR = 0.019X + 0.01	SR = 0.028X + 0.05	SR = 0.031X + 0.10	SR = 0.019X + 0.05
OVERALL PRECISION	S = 0.051X - 0.00	S = 0.047X + 0.01	S = 0.066X + 0.12	S = 0.033X + 0.05
MEAN RECOVERY	X = 0.977C - 0.01	X = 1.023C + 0.04	X = 1.055C - 0.22	X = 1.028C - 0.04
WATER 2				
SINGLE-ANALYST PRECISION	SR = 0.026X + 0.33	SR = 0.030X + 0.01	SR = 0.017X + 0.14	SR = 0.016X + 0.01
OVERALL PRECISION	S = 0.032X + 0.05	S = 0.046X + 0.05	S = 0.039X + 0.19	S = 0.017X + 0.06
MEAN RECOVERY	X = 1.020C - 0.02	X = 1.046C - 0.08	X = 1.049C - 0.11	X = 1.028C - 0.03
WATER 3				
SINGLE-ANALYST PRECISION	SR = 0.009X + 0.04	SR = 0.047X - 0.02	SR = 0.003X + 0.07	SR = 0.013X + 0.05
OVERALL PRECISION	S = 0.034X + 0.02	S = 0.082X - 0.04	S = 0.006X + 0.19	S = 0.024X + 0.05
MEAN RECOVERY	X = 1.011C - 0.01	X = 0.976C + 0.03	X = 1.050C + 0.01	X = 1.032C - 0.01

X = MEAN RECOVERY
C = TRUE VALUE FOR THE CONCENTRATION

WATER 1 - OHIO RIVER WATER
WATER 2 - MUNICIPAL SECONDARY EFFLUENT
WATER 3 - ELECTROPLATING EFFLUENT

TABLE 1 (cont.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

WEIGHTED LINEAR REGRESSION EQUATIONS FOR MEAN RECOVERY AND PRECISION (IN UG/ML)

WATER TYPE	VANADIUM, V	ZINC, ZN	SILVER, AG	BARIUM, BA
APPLICABLE CONC. RANGE	(2.25 - 99.00)	(0.10 - 1.00)	(0.10 - 4.00)	(1.00 - 20.00)
REAGENT WATER				
SINGLE-ANALYST PRECISION	SR = 0.064X + 0.15	SR =-0.009X + 0.01	SR = 0.343X - 0.03	SR = 0.067X - 0.02
OVERALL PRECISION	S = 0.109X - 0.02	S = 0.035X + 0.01	S = 0.583X - 0.05	S = 0.142X - 0.03
MEAN RECOVERY	X = 1.047C + 0.03	X = 0.984C + 0.01	X = 0.402C + 0.07	X = 0.961C + 0.05
WATER 1				
SINGLE-ANALYST PRECISION	SR = 0.045X - 0.06	SR = 0.039X + 0.02	SR = 0.221X - 0.00	SR = 0.089X - 0.05
OVERALL PRECISION	S = 0.099X - 0.12	S = 0.067X + 0.02	S = 0.765X - 0.04	S = 0.127X - 0.07
MEAN RECOVERY	X = 1.052C + 0.36	X = 0.983C - 0.06	X = 0.172C + 0.07	X = 0.970C + 0.07
WATER 2				
SINGLE-ANALYST PRECISION	SR = 0.027X + 0.01	SR =-0.022X + 0.05	SR = 0.368X - 0.03	SR = 0.197X - 0.17
OVERALL PRECISION	S = 0.128X - 0.14	S =-0.007X + 0.06	S = 0.601X - 0.05	S = 0.361X - 0.27
MEAN RECOVERY	X = 1.056C - 0.08	X = 0.959C + 0.02	X = 0.345C + 0.06	X = 0.536C + 0.64
WATER 3				
SINGLE-ANALYST PRECISION	SR = 0.011X + 0.03	SR = 0.021X + 0.02	SR = 0.979X - 0.06	SR = 0.389X - 0.42
OVERALL PRECISION	S = 0.079X - 0.04	S = 0.026X + 0.02	S = 0.956X - 0.01	S = 0.874X - 0.84
MEAN RECOVERY	X = 1.055C + 0.03	X = 0.953C + 0.00	X = 0.058C + 0.06	X = 0.329C + 0.86

X = MEAN RECOVERY
C = TRUE VALUE FOR THE CONCENTRATION

WATER 1 - OHIO RIVER WATER
WATER 2 - MUNICIPAL SECONDARY EFFLUENT
WATER 3 - ELECTROPLATING EFFLUENT

TABLE 1 (cont.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

WEIGHTED LINEAR REGRESSION EQUATIONS FOR MEAN RECOVERY AND PRECISION (IN UG/ML)

WATER TYPE	POTASSIUM, K
APPLICABLE CONC. RANGE	(4.80 - 30.00)
REAGENT WATER	
SINGLE-ANALYST PRECISION	SR = 0.012X + 0.38
OVERALL PRECISION	S = 0.080X + 0.02
MEAN RECOVERY	X = 0.923C + 0.15
WATER 1	
SINGLE-ANALYST PRECISION	SR = 0.069X + 0.00
OVERALL PRECISION	S = 0.113X - 0.10
MEAN RECOVERY	X = 0.916C + 0.01
WATER 2	
SINGLE-ANALYST PRECISION	SR = 0.055X + 0.18
OVERALL PRECISION	S = 0.107X + 0.01
MEAN RECOVERY	X = 0.921C + 0.37
WATER 3	
SINGLE-ANALYST PRECISION	SR = 0.032X + 0.36
OVERALL PRECISION	S = 0.052X + 0.45
MEAN RECOVERY	X = 0.961C - 0.06

X = MEAN RECOVERY
C = TRUE VALUE FOR THE CONCENTRATION

WATER 1 - OHIO RIVER WATER
WATER 2 - MUNICIPAL SECONDARY EFFLUENT
WATER 3 - ELECTROPLATING EFFLUENT

decreased dramatically to 20% at the 4.0 ug/mL concentration level. These findings support the conclusions of Hinners (8), EMSL-Las Vegas.

Barium recoveries were acceptable in reagent water (97%) and Ohio River Water (98%), but were poor in municipal secondary effluent (60%) and electroplating effluent (42%). It was theorized that the presence of sulfur oxides in the later two water matrices caused the barium to precipitate as barium sulfate.

For all metals, the overall standard deviations expressed as %RSD, were good. Across all water types the range was from 3% to 14% with the exceptions of silver (66%) and barium (31%). The high silver variability was observed in all four test waters while the barium %RSD was high in the municipal secondary effluent (32%) and the electroplating effluent (67%) and low in reagent water (14%) and Ohio River Water (12%).

The single-analyst standard deviations expressed as %RSD-SR, were good. Across all water types the range was from 2% to 8% with the exceptions of silver (34%) and barium (15%). The high silver variability was observed in all four test waters while the barium %RSD-SR was high in the municipal secondary effluent (17%) and electroplating effluent (29%) and low in the reagent water (6%) and Ohio River Water (8%).

Statistical comparisons were performed on all elements to determine if there were effects due to water type. Statistically significant matrix effects for calcium, cobalt, iron, manganese, molybdenum, sodium, lead, antimony, zinc, silver and barium were found to be of practical significance. The statistically significant matrix effects were not considered to be of practical significance for lead and antimony. The silver statistics were too variable to draw any conclusions.

Method detection limits (MDLs) were determined experimentally by nine laboratories on 21 elements in the four test waters. In reagent water, the experimental MDLs for barium, molybdenum, cobalt, copper, nickel, and thallium were equivalent to the SW-846 method manual MDLs (1 to 1.5 times the SW-846 MDLs). Aluminum, beryllium, calcium, iron, magnesium, antimony, vanadium, silver, cadmium, chromium, manganese, lead, and zinc had MDLs 2 to 3 times higher than those reported in the SW-846 manual. Potassium was one fifth lower and sodium was 7.5 times higher than the SW-846 MDLs. Except for potassium and sodium, the experimentally determined MDLs in this study approximated the MDLs reported in SW-846.

The analyses of an additional Youden sample pair indicated that Method 3010 could be used to analyze elements at a concentration level 50% lower than recommended in SW-846 with a decrease in recovery on the average of 3.0% and a decrease in overall and single-analyst precision on the average of 4.9% and 0.9%, respectively.

SECTION 3

RECOMMENDATIONS

SW-846 Method 3010 is recommended for the analyses of aluminum, beryllium, calcium, cadmium, cobalt, chromium, copper, iron, magnesium, manganese, molybdenum, sodium, nickel, lead, antimony, thallium, vanadium, zinc, and potassium in wastewaters. The linear regression equations obtained from this study, and presented in Table 1, can be used to predict the mean recovery, overall precision and single-analyst precision of these elements at concentrations in the ranges investigated in this study.

Statistical review of barium data revealed low recovery and poor precision (high variability) in municipal secondary effluent and electroplating effluent but good recovery and precision in reagent water and Ohio River Water. Precipitation reactions involving barium sulfate have been hypothesized for these matrix effects. Analysts are cautioned to interpret barium data carefully from any water matrix.

The mean recovery of silver was found to decrease significantly with increasing concentration in the range studied (0.1 - 4.0 ug/mL). These findings, as well as those of Hinners (8), suggests that further investigation into the optimum working range for silver analyses is recommended.

SECTION 4

DESCRIPTION OF STUDY

METHOD SUMMARY

In SW-846 Method 3010, 100 mL of sample was digested with 3 mL of concentrated nitric acid, evaporated to about 5 mL on a hot plate, then combined with 3 mL concentrated nitric acid and refluxed. After cooling, 1:1 hydrochloric acid is added. The sample is then diluted to 100 mL with Type II water. The sample is then ready for analyses of aluminum, antimony, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, molybdenum, nickel, potassium, silver, sodium, thallium, vanadium, and zinc by flame atomic absorption spectroscopy (FLAA).

STUDY DESIGN

The study design was based on Youden's non-replicate design for collaborative evaluation of an analytical method at several concentration levels over its linear range. According to Youden's design, two similar yet different samples are analyzed in pairs such that the concentration of the pairs varies between 5 and 20 percent of the mean of the pairs. For this study, six concentration levels, as three Youden pairs, were used (designated samples 1-6). The analysts were directed to do a single analysis on each sample and report one value for each element. Analyses in reagent water evaluated the proficiency of the method on a sample free of interferences; analyses in the other matrix waters were intended to reveal the effects of interferences on the method.

The primary contractor prepared the study samples by spiking measured amounts of standard solutions into the matrix waters being tested. Each spiked matrix water was then dispensed into replicate 120 mL bottles for distribution to participants. The analysts were instructed to quantitatively remove a 100 mL aliquot from each sample bottle for digestion and analysis. A matrix blank and a quality control sample, included in the study, were digested with the six samples for each water type. Background concentration levels found in the matrix blanks were subtracted from the total concentration found in each sample. Acceptance limits were provided for the quality control samples to verify that the analytical system was in control. If not in control, the problem was to be corrected and fresh aliquots of the samples, associated with that QC sample, had to be digested and reanalyzed.

Spiking solutions were provided for the MDL phase of the study. These concentrates were used to spike aliquots of the digested matrix wastes by the Method of Standard Additions.

Finally, an additional Youden sample pair, designated 7-8, was included in this study. These samples were prepared at 50% of the concentration level of samples 5-6, which were at the lowest concentration level of the optimum working range specified by SW-846. Digestion and analyses of these samples were to proceed the same as for the regular six samples.

VERIFICATION ANALYSES

To assure that the spiking solutions were properly prepared, they were analyzed using freshly prepared standards before being spiked into the test waters. A set of spiked matrix waters was digested and submitted to QARD analysts to verify sample integrity. These analyses confirmed the accuracy of the distributed samples.

SELECTION OF TEST WATERS

The four test waters selected for this study were reagent water, as the control, Ohio River Water, an electroplating company effluent and a municipal sewage treatment plant secondary effluent.

Each test water was preserved, at the time of collection, by adjusting the pH to below 2.0. Prior to dispensing, each test water was thoroughly mixed to ensure homogeneity. Aliquots were removed from each, digested according to Method 3010, then sent to QARD analysts for Inductively Coupled Plasma Spectroscopy analyses to determine the background concentrations of the 21 elements covered in this study. The results of these background screening analyses are presented in Table 2.

SELECTION OF PARTICIPATING LABORATORIES

The Quality Assurance Research Division of EMSL-Cincinnati selected the participating laboratories. As per the standard competitive bid process, an abstract of the scope of work was announced in the Commerce Business Daily. Interested laboratories were forwarded the complete request for proposal (RFP) which included the evaluation criteria upon which the offeror would be scored.

Submitted technical proposals were evaluated based upon laboratory experience and quality control practices. Laboratories whose proposals were acceptable were evaluated further in a preaward performance evaluation study. The participants selected for the formal study were the nine laboratories with acceptable proposals who performed best in the preaward study. They were as follows:

The Bionetics Corp., Hampton, VA Laboratory
J.M. Montgomery Consulting Engineers, Inc.
Spotts, Stevens and McCoy, Inc.
ERCO/ENSECO, Inc.
Thermo Analytical, Inc.
Environmental Monitoring Services, Inc.
Skinner and Sherman, Inc.
Global Geochemistry Corp.
PPB Environmental Laboratories, Inc.

TABLE 2. ICP RESULTS OF BACKGROUND SCREENING ANALYSES OF THE THREE MATRIX WATERS USED IN THE METHOD 3010 STUDY (ug/mL).

Element	Ohio River Water	Municipal Secondary Effluent	Electroplating Effluent
Al	0.140	0.136	0.071
Sb	-	-	-
Ba	0.175	0.086	0.028
Be	-	-	-
Cd	-	-	0.042
Ca	43.6	82.6	47.4
Cr	0.160	1.02	0.342
Co	1.86	7.53	-
Cu	0.232	0.202	0.034
Fe	0.319	0.553	0.891
Pb	-	-	-
Mg	11.7	19.9	7.21
Mn	0.091	0.035	0.046
Mo	-	0.079	-
Ni	0.558	1.53	0.440
K	3.40	15.8	14.6
Ag	-	-	-
Na	22.2	343	392
Tl	-	-	-
V	-	-	-
Zn	0.159	0.182	0.200

- Below instrument detection limits.

SECTION 5

TREATMENT OF DATA

The recovery and precision of SW-846 Method 3010 for 21 trace metals were determined using statistical techniques which followed ASTM Standard Practice D2777-86 (5). The statistical analyses were performed using computer programs named "Interlaboratory Method Validation Study" and hereafter referred to as IMVS (2). IMVS includes: tests for the rejection of outliers (laboratories and individual data points), summary statistics by concentration level for mean recovery, overall and single-analyst standard deviations (precision), the linear relationship between true value and mean recovery, and the linear relationship between precision and mean recovery (3), and a test of the effect of water type on recovery and precision (7). A detailed description of the statistical analytical procedures is presented below.

REJECTION OF OUTLIERS

Spurious data points are always a part of any set of data collected during an interlaboratory test program. It is important to identify and remove these data points because they can result in summary statistics which are not representative of the expected performance. However, some erratic behavior in the data may be directly related to some facet of the method itself. Therefore, spurious data points should not be removed indiscriminately, and any points that are removed should be clearly identified since further investigation of the analytical conditions related to the outliers might be of value. In some cases the data points which are deleted may actually be closer to the true concentration than the retained data. This is not inconsistent since outliers are judged relative to the average value of the data and not with respect to the true concentration. Data rejected as outliers for this study, as a result of any of the following tests for outliers, have been identified by the symbol "*" in the raw data tables.

YOUDEN'S LABORATORY RANKING PROCEDURE

In some cases the analytical values reported by a specific laboratory are so consistently high or low that a large systematic error may be attributed to that laboratory. These data are not representative of the method and should be rejected. Youden's ranking test (1) for outlying laboratories was applied separately to data from each of the waters studied. Since four water types were used in this study, the laboratory ranking procedure was applied to the four different subsets of the data at the 5% level of significance.

The Youden laboratory ranking procedure requires a complete set of data from every laboratory within a given water type. Missing data from laboratory "i" for water type "j" were replaced by the following procedure. Letting X_{ijk} denote the reported measurement from laboratory "i" for water type "j" and concentration level C_k, it is assumed that

$$X_{ijk} = \beta_j \cdot C_k^{\gamma_j} \cdot L_i \cdot \epsilon_{ijk}$$

where β_j and γ_j are fixed parameters which determine the effect of the water type "j"; L_i is the systematic error due to laboratory "i" and ϵ_{ijk} is the random within-laboratory error. Taking natural logarithms, it follows that

$$\ell n\, X_{ijk} = \ell n\, \beta_j + \gamma_j\, \ell n\, C_k + \ell n\, L_i + \ell n\, \epsilon_{ijk}$$

which is a linear regression model with dependent variable $\ell n\, X_{ijk}$ and independent variable $\ell n\, C_k$. Details and justification for this model are discussed in the section "Comparison of Recovery and Precision Across Water Types". The natural logarithms of the individual laboratory's data were regressed against the natural logarithms of the true concentration levels for the six samples in each water type. The predicted values $\ell n\, \hat{X}_{ijk}$ were obtained from the regression equation, and the missing values for X_{ijk} were estimated by $\hat{X}_{ijk} = \exp(\ell n\ \hat{X}_{ijk})$, where exp(X) denotes the constant e raised to the power X.

If the ranking test rejected a laboratory of a specific water type, then all of the laboratory's data for that water type were rejected as outliers. The rejected values were excluded from all the remaining analyses. In addition, after completion of the laboratory ranking procedure, the predicted values created to fill in for the missing data were rejected and excluded from further analyses for all laboratories.

TESTS FOR INDIVIDUAL OUTLIERS

The data remaining after the laboratory ranking procedure were grouped by water type. For each water type, the data were broken down into twenty-one subsets (one per element) defined by the six concentration levels (samples) used in the study. For each subset of data, all missing, "less than", zero, and "nondetect" data were rejected. Next, the test for individual outliers of Thompson (6), suggested in the ASTM Standard Practice D2777-86, was applied to the data using a 5% significance level. If an individual data point was rejected based on this test, it was removed from the subset, and the test was repeated using the remaining data in the subset. The test was not continued beyond the second review of the data subset.

STATISTICAL DATA SUMMARIES

Several summary statistics were calculated using the data remaining for each concentration level after the outlier rejection tests were performed. These summary statistics include: the number of retained data points, the mean recovery, recovery as a percentage of the true concentration, the absolute overall and single-analyst standard deviations, and the relative overall and single-analyst standard deviations as a percentage of the mean recovery. The basic formulas used to calculate these statistics are presented below where $X_1, X_2, \ldots, X_n$ denote the values of the n retained data points for a specific concentration level.

Mean recovery (X):

$$X = \frac{1}{n} \sum_{i=1}^{n} X_i$$

The usual concentration notation for mean recovery is $\bar{X}$. However, to be consistent with IMVS computer output, the symbol X is used in this report to represent $\bar{X}$.

Recovery as a Percent Relative Error:

$$\%\ RE = \frac{X - \text{True Value}}{\text{True Value}} (100)$$

Overall Standard Deviation:

$$S = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} (X_i - X)^2}$$

and

Percent Relative Overall Standard Deviation:

$$\%RSD = \frac{S}{X} (100)$$

The overall standard deviation, S, indicates the precision associated with measurements generated by a group of laboratories. This represents the broad variation in the data collected in an interlaboratory study. However, a measure of how well an individual analyst can expect to perform in his own laboratory is another important measure of precision. This single-analyst precision, denoted by SR, was estimated for each Youden pair by

$$SR = \sqrt{\frac{1}{2(m-1)} \sum_{i=1}^{m} (D_i - D)^2}$$

where

m = the number of complete sets of Youden pair observations remaining after outliers have been removed,

D_i = the difference between the observations in the ith Youden pair,

D = average of D_i values.

The percent relative single-analyst standard deviation was calculated by

$$\%RSD\text{-}SR = \frac{SR}{X^*}(100)$$

where X* is the average of the two mean recovery statistics corresponding to the two concentration levels defining the particular Youden pair.

These summary statistics provide detailed information on the recovery and precision of the data obtained for each concentration level. One objective of the statistical analysis of the data is to summarize the information about recovery and precision which is contained in the statistics.

A systematic relationship exists between the mean recovery (X) and the true concentration level (C) of the analyte in the sample. In addition, there are often systematic relationships between the precision statistics (S and SR) and the mean recovery (X). Usually, these systematic relationships can be adequately approximated by a linear relationship (i.e., by a straight line). Once these straight lines are established, they can be used to conveniently summarize the behavior of the method within a water type, and they can aid in comparing the behavior of the method across water types. In addition, they can be used to obtain estimates of the recovery and precision at any concentration within the applicable range studied. They can also be used to predict the behavior of the method when used under similar conditions. These important relationships are discussed on the following pages.

STATEMENT OF METHOD RECOVERY

The recovery of the method is characterized by the relationship of the mean recovery (X) to the true concentration (C) of the analyte in the water sample. In order to obtain a mathematical expression for this relationship, a regression line of the form

$$X = a + b\,C \tag{1}$$

was fitted to the data by regression techniques.

The true concentration tested covered a wide range of values. In such cases, the mean recovery statistics at the higher concentrations tend to dominate the fitted regression line producing relatively larger errors in the estimates of mean recovery at the lower concentration. To eliminate this problem, a weighted least squares technique was used to fit the mean recovery data to the true concentration values. The weighted least squares technique was performed by dividing both sides of Equation (1) by C resulting in Equation (2)

$$\frac{X}{C} = a\left(\frac{1}{C}\right) + b \tag{2}$$

The (X/C) values were regressed against the (1/C) values using ordinary least squares to obtain estimates for the values of a and b. (This is equivalent to performing a weighted least squares with weights $w = 1/C^2$; see Reference [3], page 108 for details.) Equation (2) can easily be converted to the desired relationship given by Equation (1). The intercept (b) from Equation (2) becomes the slope (b) for Equation (1) and slope (a) from equation (2) becomes the intercept (a) for Equation (1). Equation (1) can be used to calculate the percent recovery over the applicable range of concentrations used in the study.

The percent recovery is given by

$$\% \text{ Recovery} = \frac{a + bC}{C}(100) = \left(\frac{a}{C} + b\right) 100 \qquad (3)$$

If the absolute value of the ratio (a/C) is small relative to the slope (b) for concentration in the low end of the range of concentration levels used in the study, then the percent recovery can be approximated by b(100). For example, suppose the true concentration values range from 25 ug/L to 515 ug/L, the fitted line is given by $X = 0.20 + 0.85C$. The percent recovery would be approximated by 0.85 (100) = 85 percent over the specified range of 25 ug/L to 515 ug/L.

If the ratio (a/C) is not small relative to the slope (b), then the percent recovery depends upon the true concentration (C), and it must be evaluated at each concentration value within the specified range.

STATEMENT OF METHOD PRECISION

The precision of the method is characterized by the relationships between precision statistics (S and SR) and mean recovery (X). In order to obtain a mathematical expression for these relationships, regression lines of the form

$$S = d + eX \qquad (4)$$

and

$$SR = f + gX^* \qquad (5)$$

were fitted to the data by regression techniques.

As discussed previously with respect to recovery, the values of X and X* often vary over a wide range. In such cases the standard deviation statistics associated with the larger mean recovery values will dominate the regression lines. This will produce relatively larger errors in the estimates of S and SR at the lower mean recovery value. Therefore, a weighted least squares technique was also used to establish the values of the parameters d, e, f and g in Equations (4) and (5). The weighted least squares technique was performed by dividing both sides of Equation (4) by X resulting in Equation (6)

$$\frac{S}{X} = d\left(\frac{1}{X}\right) + e \tag{6}$$

and dividing both sides of Equation (5) by X* resulting in Equation (7)

$$\frac{SR}{X^*} = f\left(\frac{1}{X^*}\right) + g \tag{7}$$

The (S/X) values were regressed against the (1/X) values and the (SR/X*) values were regressed against the (1/X*) values using ordinary least squares to obtain estimates for the parameters d, e, f and g.

Equations (4) and (5) were obtained from Equations (6) and (7) in a manner similar to that discussed for mean recovery. The slope (d) for Equation (6) is the intercept (d) for Equation (4), and the intercept (e) for Equation (6) is the slope (e) for Equation (4). Similarly, the slope (f) for Equation (7) is the intercept (f) for Equation (5), and the intercept (g) for Equation (7) is the slope (g) for Equation (5).

Given equations (4) and (5), the percent relative overall standard deviation and the percent relative single-analyst standard deviation are;

$$\%RSD = \left(\frac{d}{X} + e\right)(100) \tag{8}$$

and

$$\%\text{RSD-SR} = \left(\frac{f}{X^*} + g \right) (100) \tag{9}$$

respectively. If absolute value of the ratio (d/X) is small relative to the slope (e), then the percent relative overall standard deviation can be approximated by (100)e over the applicable range of mean recovery values. Similarly if the ratio (f/X*) is small relative to the slope (g), then the percent relative single-analyst standard deviation can be approximated by (100)g over the applicable range of mean recovery values. If the ratios (d/X) and (f/X*) are not small relative to the slopes (e) and (g), then the percent relative standard deviations depend upon the values of the mean recovery statistics X and X*, and they should be evaluated separately for each value of X and X*.

COMPARISON OF RECOVERY AND PRECISION ACROSS WATER TYPES

It is possible that the recovery and precision of this method depends upon the type of water being analyzed. The summary statistics X, S and SR are calculated separately for each concentration level within each water type. They can be compared across water types in order to obtain information about the effects of water types on recovery and precision. However, the use of these summary statistics in this manner has several disadvantages. First, it is cumbersome since there are 24 mean recovery statistics (X), 24 overall precision statistics (S) and 12 single-analyst precision statistics (SR) calculated for each compound. Comparison of these statistics across concentration levels and across water types become unwieldy. Second, the statistical properties of this type of comparisons procedure are difficult to determine. Finally, due to variation associated with X, S and SR, comparisons based on these statistics can lead to inconsistent conclusions about the effect of water type. For example, reagent water may produce a significantly lower value than surface water for the precision statistics S at a high concentration, but a significantly higher value for S at a low concentration.

An alternative approach, described in reference (7), was developed to test for the effects of water type. This alternative approach summarizes the average effect of water type across concentration levels rather than studying the local effects at each concentration level. If significant differences are established by this alternative technique, then the summary statistics can be used for further local analyses.

The test for the effect of water type is based on the following statistical model. If X_{ijk} denotes the measurement reported by laboratory "i", for water type "j", and ampul "k", then

$$X_{ijk} = \beta_j \cdot C_k^{\gamma_j} \cdot L_i \cdot \varepsilon_{ijk} \qquad \begin{array}{l} i = 1,2,\ldots.,n \\ j = 1,2,\ldots.,n \\ k = 1,2,\ldots.,6 \end{array} \qquad (10)$$

The model components β_j and γ_j are fixed parameters which determine the effect of water type "j" on the behavior of the observed measurements (X_{ijk}). The parameter C_k is the true concentration level associated with ampul "k". The model component L_i is a random factor which accounts for the systematic error associated with laboratory "i". The model component ε_{ijk} is the random factor which accounts for the within-laboratory error.

The model is designed to approximate the global behavior of the data. The multiplicative structure was chosen because of two important properties. First, it allows for a possible curvilinear relationship between the data (X_{ijk}) and the true concentration level C_k through the use of the exponent γ_j on C_k. This makes the model more flexible in comparison to straight-line models. Second, as will be seen below, there is an inherent increasing relationship between the variability in the data and the concentration level C_k in this model. This property is important because it is typical of interlaboratory data collected under conditions where the true concentration levels vary widely.

Mean recovery is the expected value of the measurements (X_{ijk}). The expected value for the data modeled by Equation (10) is

$$E(X_{ijk}) = \beta_j \, (C_k^{\gamma_j}) \, E[L_i \, (\epsilon_{ijk})] \qquad (11)$$

Precision is related to the variability in the measurements (X_{ijk}). The variance of the data modeled by Equation (10) is

$$Var(X_{ijk}) = [\, \beta_j \, (\, C_k^{\gamma_j})]^2 \;\; Var[L_i \, (\epsilon_{ijk})] \qquad (12)$$

which is an increasing function of C_k.

The effect of water type on the recovery and precision of this IMVS is determined by the values of the parameters β_j and γ_j in Equations (11) and (12). If the β_j and γ_j vary with "j" (i.e., vary across water type), then the recovery and precision of the method also vary across water type.

To determine if these parameters vary across water type and to compare their values, they must be estimated from the laboratory data using regression techniques. Equation (10) represents the basic model. However, taking natural logarithms of both sides of Equation (10), the following straight-line regression model is obtained

$$\ln X_{ijk} = \ln \beta_j + \gamma_j \ln C_k + \ln L_i + \ln \epsilon_{ijk} \qquad (13)$$

which can be analyzed using standard linear model analysis techniques. The parameter $\ln \beta_j$ is the intercept and γ_j the slope of the regression line associated with water type "j". It is assumed that $\ln L_i$ is normally distributed with mean 0 and variance σ_L^2 that $\ln \epsilon_{ijk}$ is normally distributed with mean 0 and variance σ_L^2 and that the ($\ln L_i$) and ($\ln \epsilon_{ijk}$) terms are independent.

Based on Equation (13) the comparison of water types reduces to the comparison of straight lines. Reagent water is viewed as a control, and each of the remaining lines (for other water types and digestions) is compared directly to the line for reagent water.

Using the data on the log-log scale and regression techniques, the parameters $\ln \beta_j$ (and hence β_j) and γ_j can be estimated. The estimates are then used to test the null hypothesis that there is no effect due to water type. The formal null and alternative statistical hypothesis, H_0 and H_A, are given by

$$H_0: \ln \beta_j - \ln \beta_1 = 0 \text{ and } \gamma_j - \gamma_1 = 0 \qquad \text{for } j = 2,3,4$$

versus

$$H_A: \ln \beta_j - \ln \beta_1 \neq 0 \text{ and/or } \gamma_j - \gamma_1 \neq 0 \text{ for some } j = 2,3,4$$

The test of null hypothesis, H_0, against the alternative hypothesis, H_A, is based on an F-statistic derived from standard linear model theory. The probability of obtaining a value of an F-statistic as large as the value which was actually observed (F_{OBS}), denoted by $P(F>F_{OBS})$, is calculated under the assumption that H_0 is true. The null hypothesis H_0 is rejected in favor of H_A if $P(F>F_{OBS})$ is less than 0.05.

If H_0 is rejected, then some linear combination of the differences in ($\ln \beta_j - \ln \beta_1$) and ($\gamma_j - \gamma_1$) is statistically different from zero. However, this does not guarantee there will be a statistically significant direct effect attributable to any specific water type since the overall F-test can be overly sensitive to minor systematic effects common to several water types. The effect due to a specific water type is judged to be statistically significant only if one of the differences ($\ln \beta_j - \ln \beta_1$) and/or ($\gamma_j - \gamma_1$) is statistically different from zero. This is determined by checking the simultaneous 95% confidence intervals which are constructed for each of these differences. Each true difference can be stated to lie within its respective confidence interval with 95% confidence. If zero is contained within the confidence interval then there is no evidence that the corresponding difference is significantly different from zero.

If at least one of the confidence intervals for the differences ($\ln \beta_j - \ln \beta_1$) or ($\gamma_j - \gamma_1$) fails to include zero, then the statistical significance of the effect due to water type has been established. However, establishment of a statistically significant effect due to water type does not necessarily mean that the effect is of practical importance. Practical importance is related to the size and interpretation of the difference.

The interpretation of the differences involves comparing the mean recovery and standard deviation of the (X_{ijk}) data for each water type to the mean recovery and standard deviation obtained for reagent water. These comparisons are made on a relative basis. The mean recovery for water type "j" is given by Equation (11). The mean recovery for water type "j" is compared to that for reagent water (j=1) on a relative basis by

$$\frac{E(X_{ijk})}{E(X_{i1k})} = \frac{\beta_j \, C_k^{\gamma_j} \; E[L_i \, (\epsilon_{ijk})]}{\beta_1 \, C_k^{\gamma_1} \; E[L_i \, (\epsilon_{i1k})]} = \frac{\beta_j}{\beta_1} \, C_k^{(\gamma_j \, - \, \gamma_1)} \qquad (14)$$

(The ratio of the standard deviations would be equivalent to Equation (14) and therefore the interpretation of the effect on precision is the same as that for the effect on mean recovery).

The ratio in Equation (14) is a measure of the relative difference in mean recovery between water type "j" and reagent water. It is composed of two parts (a) β_j / β_1, which is independent of the true concentration level (i.e. the constant bias), and (b) $C_k^{(\gamma_j - \gamma_1)}$ which depends upon the true concentration level (i.e., the concentration dependent bias). If ($\gamma_j - \gamma_1$) is zero, then the relative difference in mean recovery is just β_j/β_1 which is independent of concentration level C_k. It can then be stated that the mean recovery of water type "j" is $100(\beta_j/\beta_1)$ percent of the mean recovery for reagent water. If ($\gamma_j - \gamma_1$) is not zero, then the mean recovery of water type "j" is $100(\beta_j/\beta_1)\ C_k^{(\gamma_j - \gamma_1)}$ percent of that for reagent water, and therefore depends upon the true concentration level C_k.

In order to illustrate these points consider the following example. Suppose that a significant F-value has been obtained, and the confidence intervals for all the differences contain zero except for water type 5. For water type 5, the point estimate for $\ln\beta_5 - \ln\beta_1$ is -0.38 and the confidence interval for $(\ln\beta_5 - \ln\beta_1)$ is (-0.69, - 0.07). The point estimate for $(\gamma_5 - \gamma_1)$ is -0.07, and the confidence interval for $\gamma_5 - \gamma_1$ is (-0.04, 0.18). In this case a statistically significant effect due to water type has been established which involves only water type 5. The practical significance of this effect is judged by considering Equation (14). The ratio of mean recoveries for water type 5 and reagent water is given by

$$\frac{E(X_{i5k})}{E(X_{i1k})} = \frac{\beta_5}{\beta_1} C_k^{(\gamma_5 - \gamma_1)} \tag{15}$$

and the ratio of the standard deviations is given by

$$\frac{Var(X_{i5k})}{Var(X_{i1k})} = \frac{\beta_5}{\beta_1} C_k^{(\gamma_5 - \gamma_1)} \tag{16}$$

Since the confidence interval for $(\gamma_5 - \gamma_1)$ contains zero this difference is assumed to be insignificant and is set to zero. Therefore Equations (15) and (16) reduce to β_5/β_1. The point estimate for $(\ln\beta_5 - \ln\beta_1)$ was -0.38. Therefore, the point estimate for β_5/β_1 is 0.68, and the mean recovery for water type 5 is estimated to be 68% of the mean recovery for reagent water. Similarly the standard deviation for the data for water type 5 is estimated to be 68% of the standard deviation for reagent water. Since the 95% confidence interval for $\ln\beta_5 - \ln\beta_1$ was (-0.69, -0.07), any value in the recovery (standard deviation) for water type 5 can be claimed to be from 50% to 93% of the mean recovery (standard deviation) for reagent water. The practical significance of the effect due to water type 5 would depend upon the importance of a mean recovery (standard deviation) observed for reagent water.

SECTION 6

RESULTS AND DISCUSSION

The primary objective of this study was to characterize the performance of SW-846 Method 3010 in terms of recovery, overall precision, single-analyst precision and the effect of water type on both recovery and precision. The statistical results for each of these performance characteristics will be discussed below.

REJECTION OF OUTLIERS

The analytical data, processed through the IMVS program, underwent three outlier tests. First, Youden's Laboratory Ranking Procedure (1) was used to detect and reject data having a large systematic error associated with a particular laboratory. If a laboratory's data for a particular element/water combination were biased either high or low, compared to the other laboratories, the laboratory failed the lab ranking procedure and all of that laboratory's data were rejected for that element/water combination. Next, zero, negative and non-detected data were rejected. Finally, the Thompson Outlier Test (6) was used to reject individual outliers. Rejected data are identified in Appendix B, Raw Data, by the symbol "*".

In this study, the IMVS program rejected a total of 995 data points or 21.9% of the 4536 data points submitted. Table 3 presents the number of rejected data for each element across water types. The maximum number of data points generated for each water type was 54 (6 Youden concentrations x 9 laboratories). The total of submitted data points for each element was 216 (54 data points per water type x 4 water types). Reagent water had the fewest rejected data, 195. The number of rejected data points was higher

TABLE 3. NUMBER OF REJECTED DATA BY ELEMENT, BY WATER TYPE AND IN TOTAL.

Element	Reagent Water	Ohio River Water	Municipal Effluent	Electroplating Effluent	Total
Aluminum	6	7	20	24	57
Antimony	4	1	7	16	28
Barium	0	15	0	6	21
Beryllium	24	22	19	23	78
Cadmium	5	12	7	8	32
Calcium	13	14	14	12	53
Chromium	7	18	18	13	56
Cobalt	15	12	13	14	54
Copper	12	13	14	12	51
Iron	13	12	14	3	43
Lead	12	12	14	8	46
Magnesium	13	12	13	14	52
Manganese	9	19	18	6	52
Molybdenum	16	15	14	9	54
Nickel	2	2	12	12	28
Potassium	12	11	13	12	48
Silver	0	12	1	24	37
Sodium	2	21	14	21	58
Thallium	8	6	1	0	15
Vanadium	8	19	14	20	61
Zinc	24	24	18	6	72
Total	195	279	258	263	995

for the other test waters but did not vary much among the waters. The number of rejected data were 279 for Ohio River Water, 258 for municipal secondary effluent, and 263 for electroplating effluent. For a single element, across all water types, the number of rejected data points ranged from 25 for thallium to 78 for beryllium. For purposes of this report, an element was considered to have excessive outliers if its total number of rejected data was equal to or greater than 54 (25% of the total submitted data). Using this criterion, the following eight elements fell into this category: aluminum (57), beryllium (78), chromium (56), cobalt (54), molybdenum (54), sodium (58), vanadium (61), and zinc (72).

The number of rejected data for each laboratory is presented in Table 4. Laboratory 6 had the highest number of rejected data with 234 while Laboratory 7 had the lowest number of rejected data with 27.

MEAN RECOVERY

Recovery was obtained by comparing the mean recovery, X, of the retained data, to the true value supplied to the IMVS program. In Appendix C the individual values for recovery are presented for each element, in each water type and at each of six concentration levels per water type. Subjecting the individual recovery values within a water type, to linear regression analysis yields regression equations where mean recovery, X, was related to the true value, C, over the entire concentration range. This reduces the 504 separate values for recovery to 84 equations; one equation for each of twenty-one trace metals in each of four water types.

The regression equations used to estimate the recovery of the 21 elements are presented in Table 1. To compare the performance of Method 3010 across water types, the recovery of each element was calculated by inserting the midpoint concentration of the range studied into the mean recovery equations given in Table 1. The estimated percent recoveries obtained for the 21 trace elements in each water type are presented in Table 5. For

TABLE 4. NUMBER OF REJECTED DATA BY LABORATORY AND OUTLIER TEST.

Laboratory	Laboratory Ranking	Thompson Outlier	Total
1	78	8	86
2	96	3	99
3	144	6	150
4	102	22	124
5	72	17	89
6	174	60	234
7	18	9	27
8	60	5	65
9	114	7	121
Total	858	137	995

reagent water the percent recoveries for the 21 trace elements were excellent, ranging from 91% to 108%, with silver (44%) being the only exception. For all waters, the recoveries ranged from 94% to 109% with barium (74%) and silver (28%) as the exceptions.

A look at the barium recovery estimates shows excellent results for reagent water (97%) and Ohio River Water (98%) but extremely poor recoveries for municipal secondary effluent (60%) and electroplating effluent (42%). It was theorized that sulfur oxides were present in the latter waters causing barium to precipitate as the sulfate.

Low silver recoveries were reported in all water types (9 - 44%). The six concentrations used in this study were 0.10, 0.15, 1.00, 1.50, 3.00 and 4.00 ug/mL. The percent recoveries of silver in reagent water are plotted against these concentrations in Figure 1 and show acceptable recovery for the low Youden pair samples, 98% and 96%, with a significant decrease in percent recovery with increasing concentration of silver. This indicates a solubility problem with silver at higher concentration levels. Hinners (8) has shown that silver in a hydrochloric acid/nitric acid mixture is soluble at a concentration of 0.05 ug/mL but forms a significant precipitate at 0.5 ug/mL.

PRECISION

The overall and single-analyst precisions were calculated for each element, in each water type and at each concentration level. This resulted in 504 individual values of overall precision (S) and 252 individual values of single-analyst precision (SR). These values are presented in Appendix C. Subjecting the individual precision values, within each water type, to linear regression analysis yields regression equations relating precision (S) or (SR) to mean recovery (X) over the entire concentration range.

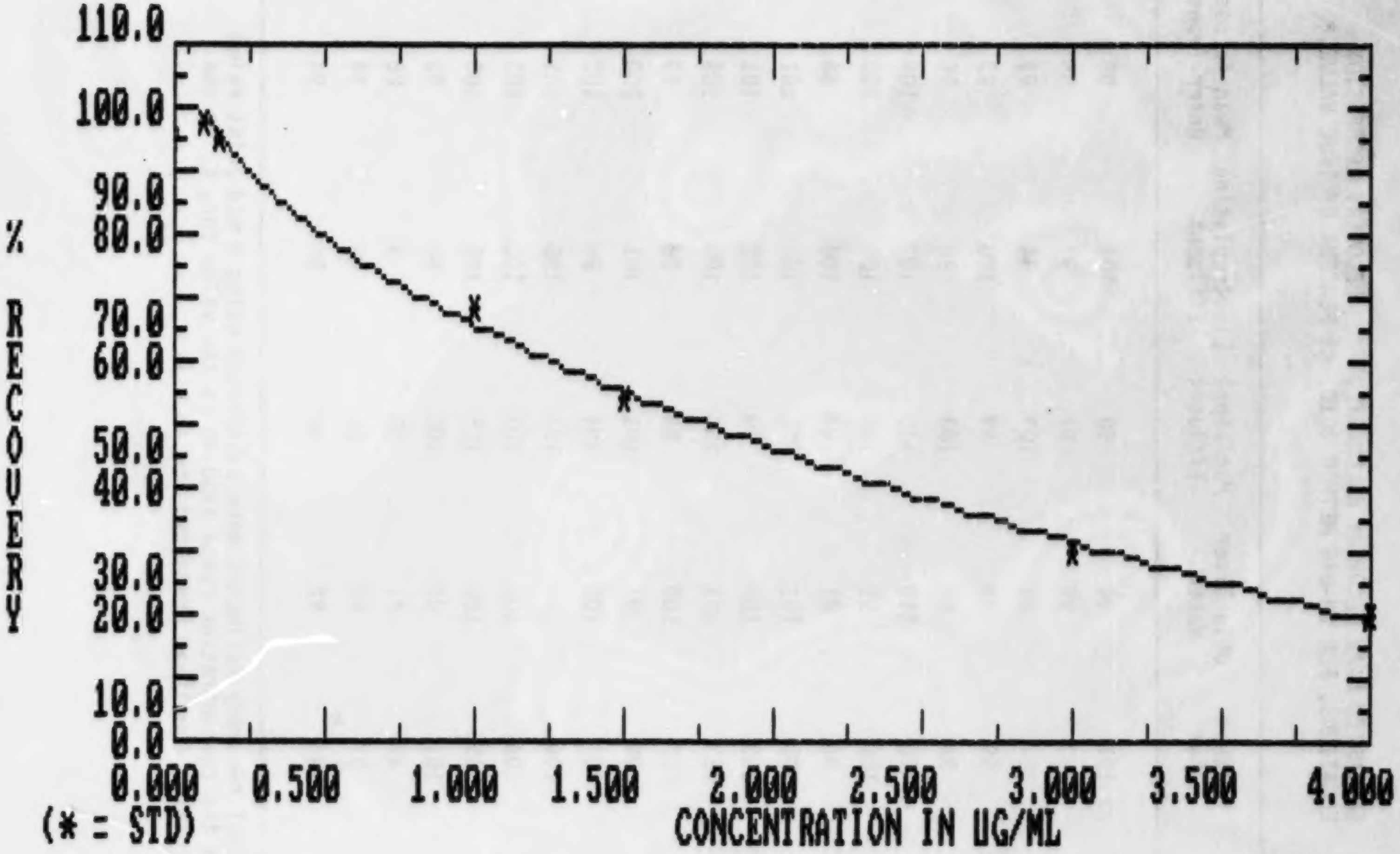
TABLE 5. ESTIMATED MEAN RECOVERY CALCULATED FROM RECOVERY REGRESSION EQUATIONS, FOR SW-846 METHOD 3010, AS PERCENT OF TRUE VALUE.*

Element	Reagent Water	Ohio River Water	Municipal Effluent	Electroplating Effluent	Mean Across Water Types
Al	101	94	98	101	98
Be	91	95	97	97	95
Ca	104	91	107	94	99
Cd	99	99	99	100	99
Co	99	98	103	98	99
Cr	108	110	110	107	109
Cu	100	99	100	100	100
Fe	96	91	96	100	96
Mg	103	101	101	101	101
Mn	103	100	99	102	101
Mo	103	101	106	100	103
Na	102	100	98	98	99
Ni	99	97	101	101	100
Pb	104	103	104	98	102
Sb	104	104	104	105	105
Tl	104	102	102	103	103
V	105	105	105	106	105
Zn	101	85	100	95	95
Ag	44	21	38	9	28
Ba	97	98	60	42	74
K	94	92	95	96	94

---

* Percent recovery estimates were calculated using a mid-point value from the concentration range studied as the value for, C, in the regression equations presented in Table 1.

FIGURE 1. RECOVERY OF SILVER AS PERCENT OF SPIKED CONCENTRATION.



The regression equations used to estimate the precision of the 21 elements are presented in Table 1. To compare the precisions across water types, the mean recovery values, calculated in Table 5, were inserted into the overall and single-analyst precision regression equations given in Table 1. Dividing the precision by the mean recovery, times 100, provides the percent relative standard deviation, %RSD or %RSD-SR.

The mean overall precision for all metals, expressed as %RSD, across water types, are presented in Table 6. The mean %RSD across water types ranged from 2.9% for thallium to 13.9% for calcium with two exceptions, barium (31.2%) and silver (65.7%). The poor silver precision was observed across all water types while the barium precision was acceptable for reagent water (13.9%) and Ohio River Water (12.0%) and poor for the municipal secondary effluent (31.6%) and the electroplating effluent (67.2%). Similar results were observed for the silver and barium mean recovery data previously discussed. With the exception of silver (52.6%) the %RSDs in reagent water ranged from 2.6% (thallium) to 13.9% (barium).

The single-analyst %RSD-SRs for the 21 elements are presented in Table 7. For all metals the mean %RSD-SR across water types ranged from 1.6% for copper to 7.6% for calcium with the exceptions of barium (15.2%) and silver (37.4%). The poor single-analyst precision for silver was observed across all water types while the barium precision was acceptable for reagent water (6.5%) and Ohio River Water (8.4%) and poor for the municipal secondary effluent (16.9%) and the electroplating effluent (28.8%). The %RSD-SRs for all elements in reagent water ranged from 1.2% (nickel) to 8.0% (calcium) with silver being the only exception at 30.9%.

EFFECTS OF WATER TYPE

The recovery and precision estimates across water types were subjected to an analysis of variance test to determine the effect water type had on the results. The probability of obtaining a value of an F-statistic as large as

TABLE 6. ESTIMATED OVERALL PRECISION BY ELEMENT AND BY WATER TYPE, AS %RSD, CALCULATED FROM THE PRECISION REGRESSION EQUATIONS IN TABLE 1.*

Element	Reagent Water	Ohio River Water	Municipal Effluent	Electroplating Effluent	Mean Across Water Types
Al	8.5	6.8	4.3	3.9	5.9
Be	11.8	5.4	4.8	3.8	6.4
Ca	11.8	14.7	14.6	14.7	13.9
Cd	2.9	3.4	5.2	3.9	3.8
Co	5.4	8.6	8.8	5.8	7.2
Cr	9.2	8.4	7.6	10.1	8.8
Cu	3.4	3.0	2.6	4.0	3.2
Fe	6.4	10.2	6.8	9.9	8.3
Mg	4.4	4.0	6.3	3.4	4.5
Mn	3.6	3.3	3.4	4.8	3.8
Mo	11.8	10.8	14.0	12.2	12.2
Na	6.6	4.0	7.4	8.3	6.6
Ni	4.1	5.1	5.2	4.2	4.6
Pb	2.8	4.8	5.1	7.8	5.1
Sb	5.4	7.2	4.8	1.5	4.7
Tl	2.6	3.8	2.3	2.9	2.9
V	10.9	9.7	12.5	7.8	10.2
Zn	5.7	11.9	12.6	7.3	9.4
Ag	52.6	66.8	53.4	89.9	65.7
Ba	13.9	12.0	31.6	67.2	31.2
K	8.2	10.4	10.8	9.1	9.6

---

* %RSDs were calculated from the regression equations presented in Table 1 using the mean recovery values generated for use in Table 5.

TABLE 7. ESTIMATED SINGLE-ANALYST PRECISION BY ELEMENT AND BY WATER TYPE, AS %RSD-SR, CALCULATED FROM THE PRECISION REGRESSION EQUATIONS IN TABLE 1.*

Element	Reagent Water	Ground Water 1	Ground Water 2	Ground Water 3	Mean Across Water Types
Al	6.2	2.4	2.6	2.3	3.4
Be	3.0	2.0	3.2	2.0	2.6
Ca	8.0	8.9	6.4	7.3	7.6
Cd	1.7	1.6	2.2	1.9	1.8
Co	2.4	2.5	4.8	2.4	3.0
Cr	3.0	3.0	3.2	3.4	3.2
Cu	1.6	2.4	1.0	1.6	1.6
Fe	3.1	5.7	3.1	4.2	4.0
Mg	2.0	2.2	6.6	1.9	3.2
Mn	2.6	1.1	1.3	2.0	1.8
Mo	7.6	5.8	5.0	4.7	5.8
Na	3.1	2.2	5.6	5.1	4.0
Ni	1.2	2.2	3.8	2.4	2.4
Pb	2.1	3.3	3.1	4.5	3.2
Sb	3.2	3.6	2.4	0.6	2.4
Tl	1.6	2.4	1.7	1.8	1.9
V	6.7	4.4	2.7	1.2	3.8
Zn	1.3	9.1	8.9	6.8	6.5
Ag	30.9	22.1	32.8	63.8	37.4
Ba	6.5	8.4	16.9	28.8	15.2
K	4.6	6.9	7.1	6.3	6.2

---

* %RSD-SRs were calculated from the regression equations presented in Table 1 using the mean recovery values generated for use in Table 5.

the value which was actually observed (F_{OBS}) is denoted by $P(F>F_{OBS})$ and was calculated under the assumption that the null hypothesis H_o (no effects) was true. The null hypotheses H_o was rejected if $P(F>F_{OBS})$ was less than 0.0500 and an effect due to water type was implied. The effect due to a specific water type was judged to be statistically significant if the 95% confidence interval constructed about either the individual intercept or slope point estimates did not contain zero. However, establishment of a statistically significant effect due to water type does not necessarily mean that the effect is of practical significance. Practical significance was determined by a review of the retained data and judging whether the statistically significant matrix effects are influenced by the retention of several errant data points or the non-uniform recovery of one of the Youden concentration pairs. If no anomalies are observed, the statistically significant matrix effect was considered to be of practical significance.

Twelve elements had $P(F>F_{OBS})$ values less than 0.0500, which implied an effect by one of the water types. With the exception of aluminum, the results of eleven elements were found to be statistically different when compared between reagent water and one of the three water types. These occurrences of statistically significant effects were reviewed to determine if the matrix effects were of practical significance. Table 8 summarizes the effect of water type. See Appendix D, Effect of Water Type, for details.

Calcium - Ohio River Water showed a statistically significant matrix effect for the intercept value. This indicates a nonuniform recovery at either the high or low concentration level. Review of the summary statistics for calcium indeed confirms this finding.

For reagent water, calcium recoveries were 101%, 102%, 101%, 110%, 106%, 113% for samples 1 to 6. For Ohio River Water, the recoveries were 94%, 95%, 90%, 86%, 89%, 87%. The precision estimates for Ohio River Water were slightly higher than for reagent water. Since the decrease in recovery occurred across several concentration levels, the statistically significant matrix effect was considered to be of practical importance and effects

TABLE 8. EFFECT OF WATER TYPE

Element	Water Type[a]	$P(F>F_{OBS})$	Statistically significant intercept at the 95% CI?	Statistically significant slope at the 95% CI?	Practical significance?
Al		.0002	---	---	---
Ca	1	.0000	yes	---	yes
Co	2	.0003	yes	yes	yes
Fe	1	.0000	yes	yes	yes
	3		yes	yes	yes
Mn	1	.0000	---	yes	yes
Mo	2	.0117	yes	yes	yes
Na	3	.0000	yes	yes	yes
Pb	1	.0002	yes	---	no
Sb	2	.0001	yes	---	no
	3		yes	yes	no
Zn	1	.0000	---	yes	yes
Ag	1	.0000	yes	---	(b)
	3		yes	yes	(b)
Ba	2	.0000	---	yes	yes
	3		---	yes	yes

(a) 1 - Ohio River Water
2 - Municipal Secondary Effluent
3 - Electroplating Effluent

(b) Data sets were too variable to draw conclusions.

calcium recovery at lower concentration levels. The precision was unaffected.

Cobalt - Municipal secondary effluent showed statistically significant matrix effects for both the intercept and slope. The recoveries for reagent water were 100%, 98%, 98%, 99%, 99%, 99% with precisions less than 7%. The recoveries for the municipal effluent were 97%, 102%, 102%, 102%, 107%, 112% with precisions ranging from 4.5% to 13.4%.

The background concentration of cobalt in the municipal effluent was 7.53 ug/mL. The concentration levels spiked into this water were 15 ug/mL, 12.5 ug/mL, 7.5 ug/mL, 6.0 ug/mL, 3.5 ug/mL, and 2.5 ug/mL. As the spike concentration levels decreased, the background concentration level had more of an influence on the recovery and precision values. The statistically significant matrix effect was considered to be of practical importance and attributable to the presence of a natural background that was about three times larger than the lowest spiked concentration.

Iron - Statistically significant matrix effects were found in both Ohio River Water and the electroplating effluent. In both cases the intercept and slope reflected the effect. The recoveries for these waters are listed below.

	Sample					
	1	2	3	4	5	6
Spike Conc. (ug/mL)	5.0	4.0	2.5	2.0	0.4	0.3
Reagent Water	96%	95%	97%	96%	94%	101%
Ohio River Water	95%	94%	90%	92%	56%	63%
Electroplating Effluent	101%	105%	101%	102%	58%	65%

The background iron concentration in Ohio River Water was 0.32 ug/mL and 0.89 ug/mL in the electroplating effluent. These background concentrations did not effect the recoveries of samples 1 through 4 but did seem to effect the recoveries for samples 5 and 6. The mean %RSD for samples 5 and 6 in Ohio River Water and the electroplating effluent were 55% and 38% compared with reagent water at 13%. The %RSDs for the other four samples, in each water type, ranged from 6% - 13%. As a result, the statistically significant matrix effects were considered of practical importance and affected the precision and recovery at lower concentrations.

Manganese - The slope estimate for Ohio River Water showed a statistically significant matrix effect compared to reagent water.

The recoveries for reagent water were 103%, 100%, 103%, 104%, 102%, 110% with precisions ranging from 1.3% to 17.9%. Ohio River Water had recoveries of 102%, 102%, 98%, 98%, 92%, 86% with %RSDs ranging from 2.0% to 23.6%. The lower recoveries for the four lowest concentration levels were not due to the retention of errant data points. Since the precision values were acceptable for the low concentration samples, the statistically significant difference in recoveries were considered to be of practical importance.

Molybdenum - Municipal secondary effluent was the only water indicating a statistically significant matrix effect for both the intercept and slope.

The recoveries for reagent water were 104%, 112%, 98%, 98%, 99%, 104% with good precision except for samples 5 and 6, 29% and 27% respectively. The recoveries for the municipal effluent were 105%, 104%, 100%, 112%, 117%, 111% with exceptable %RSDs of 6% to 13%. The recoveries between the two waters coincide for the higher concentration levels but begin to deviate at the lower levels. There was no background concentration of molybdenum in the municipal effluent. The statistically significant matrix effects for the municipal effluent are considered to be of practical importance and affect molybdenum recovery at lower concentrations levels.

Sodium - A statistically significant matrix effect was indicated for the electroplating effluent for the intercept and slope.

The recoveries for reagent water were 103%, 101%, 103%, 104%, 101%, 103%

with excellent %RSDs ranging from 6.0% to 7.4%. The electroplating effluent recoveries, 99%, 99%, 93%, 92%, 94%, 88%, were comparable for the two higher concentrations but markedly lower for the four lower concentrations. The %RSDs ranged from 5.6% to 9.1% except the lowest concentration sample had a %RSD of 23.1%. The low precision and recovery associated with sample 6 resulted from the retention of one errant data point. If removed, the recovery becomes 96% with 11.0% RSD. Even this one change does not effect the lower recoveries obtained at the lower concentration levels. The statistically significant matrix effect for the electroplating effluent was, therefore, considered to be of practical importance and affects sodium recovery at lower concentrations.

Lead - Ohio River Water showed a statistically significant matrix effect for the intercept estimate only.

The recoveries for reagent water were 103%, 102%, 103%, 103%, 100%, 100% with %RSDs from 2.0% to 12.4%. Ohio River Water recoveries were 101%, 103%, 102%, 104%, 108%, 106% with %RSDs from 4.2% to 6.9%. The statistically significant matrix effect for the intercept estimate was a result of the differences in the sample 5 and 6 recoveries in the two affected waters, 100% and 100% in reagent water and 108% and 106% in Ohio River Water. The recovery regression equations for reagent water and Ohio River Water show very little difference in slope and only a slight difference in intercept. Using these equations to calculate mean recovery results in less than 1% difference between the recovery estimates. Additionally, the overall precision of the data sets for these two water matrices are very good. As a result, the statistically significant matrix effect observed for lead is found to be of no practical significance.

Antimony - Antimony showed matrix effects for the municipal secondary effluent and the electroplating effluent. The recoveries for these two waters are compared to reagent water below.

	Sample					
	1	2	3	4	5	6
Reagent Water	108%	106%	102%	104%	81%	92%
Municipal Secondary Effluent	105%	107%	104%	102%	99%	96%
Electroplating Effluent	105%	105%	104%	105%	109%	104%

The %RSDs for reagent water were 1.4% to 8.7% for samples 1 to 4 and 19% and 23% for samples 5 and 6. The %RSDs for municipal secondary effluent were 3.2% to 6.3% for samples 1 to 4 and 14% and 22% for samples 5 and 6. The %RSDs for the electroplating effluent were 1.1% to 2.0% for samples 1 to 4 and 13% and 15% for samples 5 and 6. The %RSDs of these three waters, including samples 5 and 6, were very similar indicating that the recoveries among water types, at the lower concentration levels, were indeed different. However, because it is the reagent water samples that are different, compared to the other matrix waters, practical significance cannot be established.

Zinc - Zinc had a statistically significant matrix effect for Ohio River Water for the slope value only. This suggests a uniform difference in recoveries across all concentration levels. The recoveries for reagent water were:

	Sample					
	1	2	3	4	5	6
Reagent Water	100%	99%	98%	98%	107%	102%
Ohio River Water	98%	90%	84%	89%	58%	35%

The %RSDs for the reagent water were very good from 3.6% to 11.7%. Ohio River Water, however, had more variable data with %RSDs ranging from 4.3% to 85%.

The statistically significant matrix effect for Ohio River Water is considered to be of practical importance and effects zinc recovery and precision across all concentration levels.

Silver - The poor behavior of silver in this study has been previously discussed under Mean Recovery. Because of the insolubility problems encountered with the four highest concentration levels a comparison between water types would not provide any useful information.

Barium - Barium showed statistically significant matrix effects for both the municipal secondary effluent and the electroplating effluent. A comparison of the recoveries for these waters follows.

	Sample					
	1	2	3	4	5	6
Reagent Water	92%	97%	98%	100%	102%	100%
Municipal Secondary Effluent	38%	45%	73%	86%	112%	110%
Electroplating Effluent	21%	24%	48%	76%	110%	110%

The %RSDs were the following:

	%RSD					
	1	2	3	4	5	6
Reagent Water	18%	12%	12%	13%	9%	13%
Municipal Secondary Effluent	56%	43%	12%	11%	14%	6%
Electroplating Effluent	61%	114%	73%	45%	14%	16%

The background concentration of barium in the municipal effluent was 0.086 ug/mL and for the electroplating effluent was 0.028 ug/mL. The concentration levels spiked into these waters were 20 ug/mL, 16 ug/mL, 8.0 ug/mL, 6.0 ug/mL, 1.2 ug/mL, and 1.0 ug/mL. No background screening analyses were performed to determine the presence of anions. It is possible that elevated sulfate levels were present in these two effluents which caused precipitation of the barium during the digestion procedure. The acceptable performance of barium in reagent water and Ohio River Water reinforce the statistically significant matrix effects observed in the two effluents. These effects were considered to be of practical importance and effect barium recovery and precision above approximately 2.0 ug/mL.

METHOD DETECTION LIMITS (MDLs)

Method detection limits (MDLs) were experimently determined in reagent water using the procedure described in the Introduction Section of this report. The reported MDL data from the nine participating laboratories are presented in Appendix E. Some laboratories reported an MDL of zero which were not included in the average MDLs calculated.

The average MDL for each element in reagent water is presented in Table 9 and is compared with the MDLs found in the SW-846 method manual. This comparison shows that the MDLs generated in this study approximate those reported in SW-846 for the following elements: barium, molybdenum, cobalt, copper, nickel, and thallium which were only 1 to 1.5 times higher than the SW-846 MDLs. The MDLs for aluminum, beryllium, calcium, iron, magnesium, antimony, vanadium, silver, cadmium, chromium, manganese, lead, and zinc were 2 to 3 times higher than the SW-846 MDLs. The MDL for potassium was one fifth of the SW-846 MDL while the MDL for sodium was 7.5 times higher than the SW-846 MDL.

EVALUATION OF LOWEST CONCENTRATION LEVELS

The final objective of this study was to verify that the lowest concentration levels specified in the SW-846 method manual were obtainable. To accomplish this objective an additional sample pair, designated 7-8, were included in the study at a concentration level 50% lower than samples 5-6, which were at the lowest concentration levels specified in SW-846.

The comparisons of the mean recoveries and the net changes for the 21 elements in reagent water for samples 5-6 versus samples 7-8, are presented in Table 10. The concentration values and recoveries shown in Table 10 are the averages of the two values comprising each sample pair. The average percent recovery for the sample at 50% concentration level were only 3.0% lower than the average percent recovery at the lowest concentration level specified in the SW-846 method manual. Twelve elements: aluminum, calcium, cobalt, chromium, copper, iron, magnesium, sodium, nickel, lead, vanadium,

TABLE 9. COMPARISON OF MEASURED METHOD DETECTION LIMITS (MDLs) VERSUS SW-846 METHOD DETECTION LIMITS (ug/L).

Element	SW-846 MDLs	Measured MDLs in Reagent Water
Al	100	299
Ba	100	142
Be	5	9
Ca	10	29
Fe	30	60
K	10	2
Mg	1	3.5
Mo	100	149
Na	2	15
Sb	200	378
V	200	356
Ag	10	22
Cd	5	16
Co	50	60
Cr	50	96
Cu	20	18
Mn	10	22
Ni	40	46
Pb	100	239
Tl	100	114
Zn	5	11

* Average results from nine laboratories.

TABLE 10. COMPARISON OF THE RECOVERY IN REAGENT WATER OF SAMPLES 5 & 6 VERSUS SAMPLES 7 & 8, AS PERCENT OF TRUE VALUE[a]

	Sample 5 & 6		Sample 7 & 8		
Element	Mean Conc.[b] (ug/mL)	Mean %[c] Recovery	Mean Conc.[b] (ug/mL)	Mean %[c] Recovery	% Difference
Aluminum	5.5	95.8%	2.75	92.7%	-3.1
Antimony	1.4	86.2%	0.68	78.5%	-7.7
Barium	1.1	98.9%	0.56	97.3%	-1.6
Beryllium	0.05	92.6%	0.025	85.3%	-7.3
Cadmium	0.05	103.2%	0.025	108.6%	5.4
Calcium	21.0	108.6%	10.5	103.7%	-4.9
Chromium	0.55	102.9%	0.27	101.1%	-1.8
Cobalt	3.0	98.2%	1.25	99.1%	0.9
Copper	0.22	101.9%	0.12	102.7%	0.8
Iron	0.35	95.5%	0.18	93.4%	-2.1
Lead	1.0	100.3%	0.50	100.9%	0.6
Magnesium	6.0	102.2%	2.5	102.3%	0.1
Manganese	0.12	105.7%	0.06	95.9%	-9.8
Molybdenum	1.4	107.7%	0.70	88.1%	-19.6
Nickel	0.35	93.6%	0.175	89.5%	-4.1
Potassium	5.4	96.0%	2.70	90.0%	-6.0
Silver	0.12	96.7%	0.06	114.1%	17.4
Sodium	108	102.0%	30	105.8%	3.8
Thallium	1.1	101.0%	0.54	84.3%	-16.7
Vanadium	2.5	105.2%	1.21	103.8%	-1.4
Zinc	0.12	104.2%	0.055	97.2%	-7.0
				Average Deviation	-3.0

(a) Percent recovery data was taken from reagent water sample set.

(b) Arithmetic mean of the sample pair concentrations.

(c) Arithmetic mean of the sample pair percent recoveries.

and barium had less than ± 5% net change in recovery. Six elements: beryllium, cadmium, manganese, antimony, zinc, and potassium had a ± (5%-10%) net change. For molybdenum and thallium, the 50% concentration level was 19.6% and 16.7% lower for the mean recovery, respectively, than the lowest concentration level specified in SW-846. Silver had a mean recovery 17.4% higher for the 50% concentration level samples when compared to the specified concentration level recoveries.

The overall %RSDs for samples 5-6 versus samples 7-8 are compared in Table 11 for the 21 elements in reagent water. The overall %RSDs for samples 7-8 were slightly more variable than samples 5-6. The average overall %RSD for the samples at 50% concentration level were 4.9% higher than the average overall %RSD of the lowest concentration level samples specified in SW-846. Fourteen elements: aluminum, calcium, cobalt, chromium, copper, iron, magnesium, molybdenum, sodium, lead, vanadium, zinc, barium, and potassium had a net change less than ± 5%. Five elements: beryllium, cadmium, manganese, nickel, and antimony had a net change between ±(5% - 10%). Only silver (14.0%) and thallium (26.9%) had a net change greater than ± 10%.

The comparison of the single-analyst %RSD-SRs for samples 7-8 versus samples 5-6 are presented in Table 12. The average single-analyst precisions for the 50% concentration level samples were 0.9% higher than the lowest concentration level specified in SW-846. Twelve elements: aluminum, antimony, barium, beryllium, cadmium, cobalt, copper, magnesium, potassium, silver, sodium, and thallium had net changes less than ± 5%. Seven elements: calcium, chromium, lead, manganese, nickel, vanadium, and zinc had net changes between ± (5% and 10%) while iron (11.8%) and molybdenum (15.4%) were the only elements with net changes greater than ± 10%.

In summary, SW-846 Method 3010 can be used to analyze elements at concentration levels 50% lower than recommended by SW-846 with a decrease in percent recovery on the average of 3.0% and a decrease in overall and single-analyst precision on the average of 4.9% and 0.9%, respectively.

TABLE 11. COMPARISON OF OVERALL PRECISION IN REAGENT WATER, AS % RSD, OF SAMPLES 5 & 6 VERSUS SAMPLES 7 & 8.(a)

	Samples 5 & 6		Samples 7 & 8		
Element	Mean Conc.(b) (ug/mL)	Mean(c) %RSD	Mean Conc.(b) (ug/mL)	Mean(c) %RSD	% Difference
Aluminum	5.5	8.0%	2.80	8.9%	0.9
Antimony	1.4	21.3%	0.68	28.5%	7.2
Barium	1.1	11.3%	0.56	11.2%	-0.1
Beryllium	0.05	18.0%	0.025	24.7%	6.7
Cadmium	0.05	6.4%	0.025	17.1%	10.7
Calcium	21.0	11.2%	10.5	11.8%	0.6
Chromium	0.55	10.8%	0.27	13.9%	3.1
Cobalt	3.0	3.7%	1.25	4.5%	0.8
Copper	0.22	5.6%	0.12	7.7%	2.1
Iron	0.35	13.6%	0.18	9.9%	-3.7
Lead	1.0	10.9%	0.5	15.3%	4.4
Magnesium	6.0	1.4%	2.5	3.7%	2.3
Manganese	0.12	12.1%	0.06	22.6%	10.5
Molybdenum	1.4	24.4%	0.70	26.5%	2.1
Nickel	0.35	12.4%	0.18	22.4%	10.0
Potassium	5.4	7.9%	2.7	10.5%	2.6
Silver	0.12	13.2%	0.06	27.2%	14.0
Sodium	108	6.6%	30	5.8%	-0.8
Thallium	1.1	6.7%	0.54	33.6%	26.9
Vanadium	2.5	8.6%	1.2	10.8%	2.2
Zinc	0.12	9.7%	0.055	9.9%	0.2
				Average Deviation	4.9

(a) Percent RSD data was taken from reagent water sample set.

(b) Arithmetic mean of the sample pair concentrations.

(c) Arithmetic mean of the sample pair percent RSD's.

TABLE 12. COMPARISON OF SINGLE-ANALYST PRECISION IN REAGENT WATER, AS %RSD-SR, OF SAMPLES 5 & 6 VERSUS SAMPLES 7 & 8. (a)

	Samples 5 & 6		Samples 7 & 8		
Element	Mean Conc.(b) (ug/mL)	Mean(c) %RSD-SR	Mean Conc.(b) (ug/mL)	Mean(c) %RSD-SR	% Difference
Aluminum	5.5	9.5%	2.75	7.2%	-2.3
Antimony	1.4	18.3%	0.68	17.2%	-1.1
Barium	1.1	5.4%	0.56	9.0%	3.6
Beryllium	0.05	16.7%	0.025	15.4%	-1.3
Cadmium	0.05	4.1%	0.025	3.9%	-0.2
Calcium	21.0	5.1%	10.5	13.4%	8.3
Chromium	0.55	12.1%	0.27	6.5%	-5.6
Cobalt	3.0	1.5%	1.25	1.5%	0
Copper	0.22	5.0%	0.12	4.5%	-0.5
Iron	0.35	16.8%	0.18	5.0%	11.8
Lead	1.0	4.7%	0.5	10.6%	5.9
Magnesium	6.0	1.7%	2.5	5.2%	3.5
Manganese	0.12	6.6%	0.06	12.5%	5.9
Molybdenum	1.4	24.8%	0.70	9.4%	-15.4
Nickel	0.35	7.7%	0.18	14.9%	7.2
Potassium	5.4	8.0%	2.7	5.9%	-2.1
Silver	0.12	6.6%	0.06	9.0%	2.4
Sodium	108	2.0%	30	2.5%	0.5
Thallium	1.1	6.6%	0.54	5.5%	-1.1
Vanadium	2.5	2.8%	1.2	9.4%	6.6
Zinc	0.12	10.0%	0.055	3.0%	-7.0
				Average Deviation	0.9

(a) Percent RSD-SR data was taken from the reagent water sample set.

(b) Arithmetic mean of the sample pair concentrations.

(c) Arithmetic mean of the sample pair percent RSD-SR's.

REFERENCES

1. Youden, W.J. and Steiner, E.M. Statistical Manual of the AOAC, Association Of Official Analytical Chemists, Arlington, Virginia, p. 88 (1975).

2. Outler, E.C. and McCreery, J.H. Interlaboratory Method Validation Study: Program Documentation. Battelle Columbus Laboratories. 1982, USEPA Contract No. 68-03-2624.

3. Draper, N.R. and Smith, H. Applied Regression Analysis, 2nd Edition, John Wiley and Sons, New York, 1981.

4. Winefordner, J. D. and Long, G. L., "Limit of Detection", Analytical Chemistry, Vol. 55, No. 7, June 1983.

5. Annual Book of ASTM Standards, Part 31, Water, American Society for Testing Materials, Philadelphia, Pennsylvania, ASTM D2777-86, "Standard Recommended Practice for Determination of Precision and Bias of Methods of Committee D-19 on Water."

6. Thompson, W.R., "On a Criterion for the Rejection of Observations and the Distribution of the Ratio of the Deviation to the Sample Standard Deviations", Annals of Mathematical Statistics, Vol. 6, 1935, p. 214.

7. Bishop, T., Brydon, F. and Outler, E.C. Battelle Report for EPA Contract 68-03-2624, Development of Statistical Technique to Compare Analytical Methods Across Wastewaters.

8. Hinners, T.A., "Stability of Silver Solutions Using Procedures Specified by EPA", Draft Report, USEPA, Environmental Monitoring Systems Laboratory-Las Vegas.

APPENDIX A

LABORATORY INSTRUCTIONS
AND
SW-846 METHOD 3010

1. INTRODUCTION

The purpose of this Interlaboratory Method Validation Study (IMVS) is to provide precision and accuracy statements for 21 metals in aqueous samples and reagent water for Resource Conservation and Recovery Act (RCRA) 846 analytical method 3010. The 21 metals of interest are aluminum (Al), barium (Ba), beryllium (Be), calcium (Ca), cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), iron (Fe), lead (Pb), potassium (K), magnesium (Mg), manganese (Mn), molybdenum (Mo), sodium (Na), nickel (Ni), antimony (Sb), vanadium (V), zinc (Zn), silver (Ag), and thallium (Tl).

The enclosed samples are split into two mixes, MIX 1 containing 18 elements and MIX 2 containing three.

Mix 1			Mix 2
Al	Cr	Mo	Ag, Ba, K
Cu	Na	Be	
Fe	Ni	Ca	
Pb	Cd	Mg	
Sb	Co	Mn	
V	Tl	Zn	

Each mix has already been spiked into all aqueous matrices for this study. No further spiking will be necessary.

The design of the IMVS is based on Youden's original non-replicate design for collaborative evaluation of precision and accuracy for analytical methods. According to Youden's design, two similar yet different samples are analyzed in pairs. For the IMVS of 3010 four Youden pairs will be used thus totally eight samples per water type. The analyst is directed to do a single analysis and report one value for each sample.

OPTION II and OPTION IV of the Request for Proposal (RFP) are also being exercised for this study. The instructions for these options will be covered in later sections of this booklet. Also, you will find several bottles labelled SPEICAL. These bottles are to be digested only and returned to Bionetics Corporation with the empty coolers. Instructions for these bottles will also be covered in later sections of this booklet.

Each contractor has 60 calendar days from receipt of the samples to complete all analytical work. The address to which all data is to be returned is covered in section 4 Data Report Forms.

A laboratory notebook is being provided to each contractor for the collection of all raw data and analytical procedure descriptions. The yellow tear out pages are for the contractor to keep. These notebooks must be returned to The Bionetics Corporation at the completion of the study.

---

1. Youden, W.J. Statistical Techniques for Collaborative Tests. Association of Official Analytical Chemists, Inc., Washington, D.C., 1969. 64 pp.

2. ANALYTICAL PROCEDURES

Before proceeding, make sure you have all the necessary sample bottles as outlined on the packing list.

2.1 Sample Digestion

Shake all bottles well before removing aliquots.

Each bottle contains approximately 110 milliliters of spiked aqueous sample or reagent water. From each bottle quantitatively transfer a 100 milliliter aliquot to a digestion beaker and proceed with the digestion procedure as outlined in section 7.1 of method 3010. Digest all samples, as a group, according to water type. Table II summarizes the 3010 sample groupings. A typical digestion group will be as follows.

8 Samples (e.g. Mix 1, WATER 1, Amp 1 through Amp 8).
2 QC Samples (ICAP-19 and ICAP-7).
1 Matrix blank (e.g. WATER 1).
1 Procedural blank (Laboratory reagent water).

QC sample and blank digestions are covered in subsequent sections.

Record all digestion procedures in the laboratory notebook provided. This will include exact volumes and type of acid added and the duration of heating cycle and hot plate settings throughout each digestion group. It is important that method 3010 be followed exactly for this is a method validation study not a method development study. Do not attempt to maximize any step in the digestion procedure based on past experience. Failure to follow the prescribed method can lead to nonpayment or forfeiture of your contract.

After the samples have been digested dilute the digestate to 100 milliliters using ASTM type II water. The sample is now ready for analysis. Total sample digestions = 64.

2.2 Blank Digestion

Two types of blanks will be used for this study. The first is a matirx blank and the second a procedural blank.

For the matrix blanks three aqueous matrices and one reagent water matrix are provided in one gallon bottles. These matrices are unspiked and require digestion and analysis for background concentrations. Remember, the appropriate matrix blank must be digested with each sample group containing that matrix. These matrix blank values will be used to correct the total element concentrations for background in both the regular study samples and the OPTION IV Digestates. Number of matrix blanks to be digested are 4 matrices x 2 mixes = 8.

The procedural blank will be your own laboratory reagent water digested with each of the 3010 sample groups using the same reagents and procedures. These blanks will be used to isolate systematic errors which may be introduced into the results based on laboratory procedure. A procedural blank should be analyzed with each sample group. Number of procedural blanks to be digested = 8.

The digestion procedure will be the same as described for the samples. Each blank matrix must be well shaken before aliquots are removed. Remove a 100 milliliter aliquot from each matrix and digest according to the method 3010 section 7.1. Record all digestion conditions in the laboratory notebook. When completed dilute the blank digestates to 100 milliliters using ASTM type II water. The blanks are now ready for analysis.

2.3 Quality Control Sample Digestions

Twenty U.S. EPA Quality Control Samples are being provided for this study. There are 10 U.S. EPA ICAP-19 WP 1083 conc 1 ampuls and 10 U.S. EPA ICAP-7 WP 1083 conc 1 ampuls. Dilution instructions are as follows.

ICAP-19	Pipet exactly 20 mL into a 100 mL volumetric flask. Bring to volume with ASTM type II water.
ICAP-7	Pipet exactly 5 mL into a 100 mL volumetric flask. Bring to volume with ASTM type II water.

The reference values for these diluted QC samples, and the ± 20% acceptance limits, are found in Table 13.

Table 13

Reference Values for QC Samples

ICAP-19

Parameter	Reference Value (mg/L)	Acceptance Limits (mg/L)	
		80%	120%
Sb	1.98	1.58	2.38
Be	1.92	1.54	2.30
Cd	1.88	1.50	2.26
Ca	2.00	1.60	2.40
Cr	2.06	1.65	2.47
Cu	2.06	1.65	2.47
Co	2.00	1.60	2.40
Fe	2.04	1.63	2.45
Pb	2.02	1.62	2.42
Mg	2.00	1.60	2.40
Mn	2.04	1.63	2.45
Mo	2.12	1.70	2.54
Ni	2.04	1.63	2.45
Tl	2.02	1.62	2.42
V	2.02	1.62	2.42
Zn	2.02	1.62	2.42

ICAP-7

Parameter	Reference Value (mg/L)	Acceptance Limits (mg/L)	
		80%	120%
Ag	4.95	3.96	5.94
Al	4.85	3.88	5.82
Ba	4.85	3.88	5.82
K	50.0	40.0	60.0
Na	4.30	3.44	5.16

The quality control samples are now ready for digestion. Quantitatively transfer 100 milliliters of each of the QC samples to digestion beakers and proceed with the digestion procedure outlined in method 3010 section 7.1. Please note, one ICAP-19 and one ICAP-7 QC sample must be digested with each Mix 1 sample group. These QC samples are being used as independent check samples on the entire analytical procedure, from digestion to instrumental analysis. If an analyte value falls outside of its acceptance limits the entire group of samples will have to be redigested and analyzed at the participating laboratory's expense. Additional sample bottles and QC ampuls can be obtained by calling Mr. Kenneth Edgell at the Bionetics Corporation (513) 771-0448. Be extremely cautious during all digestion procedures.

A quality control sample report form (green form) has been included with this study for the reporting of QC results. This form is also located in the back of this booklet.

3. INSTRUMENTAL ANALYSIS

Instrumental analyses will be performed using conditions and methods specified in the EPA 7000 series of methods and your instrument manual.

The number and type of samples to be analyzed for the method validation study are summarized in TABLE II. A total of 64 samples (exclusive of blanks and quality control samples) must be digested and analyzed.

Within each sample group the analytical order will be from Amp 8 (lowest concentration) to Amp 1 (highest concentration). The minimum analytical outline to be followed will be the following.

Calibration curve - 8 water types (Amp 8 through Amp 1) - 1 matrix blank - 2 quality control samples (ICAP-19 & ICAP-7) - 1 procedural blank - calibration check sample.

This minimum outline should not preclude your normal QC/QA procedures.

Record all raw data obtained in the laboratory notebook.

4. DATA REPORT FORMS

There are three sets of data report forms contained in this instruction booklet. The main set (white pages) are the forms to use for the study 3010 results. A second set (yellow pages) contains report forms for the OPTION IV results, the third set (blue pages) contains the OPTION II report form and the final set (green pages) contain the quality control report forms and instrument identification form.

The data report forms have been bound into this booklet for easier use. Each report form is devoted to one element for several water types. Close attention should be paid to the form you are filling out.

For all bottles marked Amp 1 through Amp 6 an analytical value MUST be reported. Contractors will not be paid for data reported as zero or less than MDL. There is sufficient analyte present in each of these bottles to make analytical data obtainable by all contractors. This requirement also applies to the OPTION IV digestate samples.

There are exceptions to this requirement. All bottles labelled AMP 7 and AMP 8 can be reported as zero or less than an MDL. The analyte concentrations in AMP 7 and AMP 8 are very low and will require an optimally tuned instrument to obtain data from some of the sample waters.

Several of the standard additions concentrations in OPTION II may not be analyzable on your instrument. It is acceptable to report a zero or less than an MDL for OPTION II data calculations.

Make sure your laboratory code is on each page. All data is to be reported as milligrams per liter (mg/L) and corrected for background concentrations. Numbers are to be right justified in the appropriate boxes. A decimal point will have its own box.

Ampule 1

			1	2	.	2

1 - 7

The significant figure format to be used is as follows.

XXX
XX.X
X.XX
0.XX
0.0XX

At the completion of this study, return the laboratory notebook and data report form booklet to:

Mr. Kenneth Edgell
The Bionetics Corporation
16 Triangle Park Drive
Cincinnati, Ohio 45246
(513) 771-0448

5. QUALITY CONTROL/QUALITY ASSURANCE REQUIREMENTS

Each participating laboratory will be required to implement quality control/quality assurance protocols for this study. These protocols must include, but are not limited to, the following procedures:

1. Preparation of calibration curves for each element. Each curve will consist of a blank and at least four calibration standards.
2. Analysis of check standards at the beginning and end of each working day and at a frequency of 10% during analytical runs. If analysis of a check standard is not within ± 5% of the expected value, analysis must be terminated, the instrument must be recalibrated, and all samples run since last successful check standard must be reanalyzed.
3. Preparation and analysis of one procedural blank for every water group.
4. Analysis of an outside reference standard having known values. This sample will be provided by EPA and will be analyzed with each analytical group as covered in section 2.3 of this packet.
5. Maintenance of accurate written records that trace all samples from receipt through preparation, analysis, and final data submittal. Each contractor will be required to maintain all pertinent raw and final data until informed otherwise by EPA.
6. Maintenance of logs documenting instrument repairs or modifications performed during the time frame of this study.

6. OPTION II

Option II is the determination of the AA-flame Method Detection Limit (MDL) for each of the elements specified in each of the four matrices. Each Contractor shall determine the MDL's by the following procedure:

> The detection limit for each of the specified elements shall be evaluated by first, determining the average noise level of response, seven (7) times for a single reagent water (laboratory Type II water) digestate with no detectable elements. The mean and standard deviation of the noise level is calculated. Second, for each of the four matrix blank digestates a calibration curve shall be developed using the method of standard additions (five points). The slope of each calibration curve, m, shall be calculated by using linear regression. The MDL shall be calculated by using the following relationship.
> MDL = K(S/m)
>
> K = 3; MDL = method detection limit(ug/mL).
> S = standard deviation of the noise level (as absorbance units).
> m = regressed slope from standard additions curve(absorbance units/ug/mL).

The standard deviation of the noise level is to be calculated as absorbance units. Do no convert these values to concentration units. The slope of the standard additions curve is to be calculated as absorbance units/concentration.

Ampul concentrates provided are to be used as spiking solutions for the standard additions procedure. There are two ampuls labelled conc 1 and conc 2. Both ampuls are to be diluted in the following manner.

OPTION I Conc 1	Pipet 20 mL into a 200 mL volumetric flask. Bring to volume with ASTM type II water.
OPTION I Conc 2	Pipet 20 mL into a 200 mL volumetric flask. Bring to volume with ASTM type II water.

The elements and concentrations in each diluted flask are listed on the following page.

OPTION II conc 1

Element	Concentration
Al	9.60 ug/ml
Ba	9.60 ug/ml
Be	0.51 ug/ml
Ca	0.96 ug/ml
Fe	3.20 ug/ml
K	0.96 ug/ml
Mg	0.096 ug/ml
Mo	9.60 ug/ml
Na	0.23 ug/ml
Sb	20.4 ug/ml
V	21.2 ug/ml

OPTION II conc 2

Element	Concentration
Ag	0.96 ug/ml
Cd	0.48 ug/ml
Co	4.80 ug/ml
Cr	4.80 ug/ml
Cu	2.00 ug/ml
Mn	0.96 ug/ml
Ni	4.00 ug/ml
Pb	9.60 ug/ml
Tl	9.60 ug/ml
Zn	0.56 ug/ml

There is sufficient matrix blanks provided for digestion and use in this MDL study.

The method of standard additions outline to be used for each for the four matrices is listed below.

Standard Additions Outline

Matrix Digestate	Spike Solution	Reagent Water	Total
25 mL	1 mL	24 mL	50 mL
25 mL	2 ml	23 mL	50 mL
25 mL	5 mL	20 mL	50 mL
25 mL	10 mL	15 mL	50 mL
25 mL	20 mL	5 mL	50 mL

Because several of the aqueous matrices have high background concentrations, not all metals will be analyzed in all matrices. TABLE shows which elements will be analyzed, and in what matrix, for OPTION II.

ALL OPTION II absorbances should be recorded in the laboratory notebook.

The data report forms for OPTION II results are found in the data report form booklet (green forms). Report data as milligrams per liter (mg/L). Reporting of zeroes or less than an MDL is allowed for this option. If there is no noise level to calculate a mean and standar deviation than just report zeroex in those spaces and proceed with the standard additions curve.

OPTION II Element vs Matrix list

Conc 1

Element	Water 1	Ohio Water 2	MSD Water 3	Varland Water 4
Al	*	*	*	*
Ba	*	*	*	*
Be	*	*	*	*
Ca	*	Do not run	Do not run	Do not run
Fe	*	Do not run	Do not run	Do not run
K	*	Do not run	Do not run	Do not run
Mg	*	Do not run	Do not run	Do not run
Mo	*	*	*	*
Na	*	Do not run	Do not run	Do not run
Sb	*	*	*	*
V	*	*	*	*

Conc 2

Element	Water 1	Water 2	Water 3	Water 4
Ag	*	*	*	*
Cd	*	*	*	*
Co	*	Do not run	Do not run	*
Cr	*	*	Do not run	*
Cu	*	*	*	*
Mn	*	*	*	*
Ni	*	*	Do not run	*
Pb	*	*	*	*
Tl	*	*	*	*
Zn	*	Do not run	Do not run	Do not run

* Analyze element in this water

7. OPTION IV

Under this option the contractor shall analyze EPA supplied digestate samples (two mixes) for each of four matrices. ALL OPTION IV bottles have green labels which clearly state OPTION IV DIGESTATE.

These samples have been digested and brought to a final volume of 100 mL. These samples are ready to analyze. Each contractor should use their matrix blank values, previously digested and analyzed, for correction of OPTION IV data. Dilute an ICAP-19 and ICAP-7 quality control sample as per the instructions given in section 2.3 of this manual. No digestion is necessary, they are ready to use. The QC acceptance limits are the same as found in TABLE III.

METHOD 3010

ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS FOR ANALYSIS BY FLAA OR ICP SPECTROSCOPY

1.0 SCOPE AND APPLICATION

1.1 This digestion procedure is used for the preparation of aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for analysis, by FLAA or ICP, for the metals listed below. The procedure is used to determine total metals.

1.2 Samples prepared by Method 3010 may be analyzed by FLAA or ICP for the following:

Aluminum
Antimony
Barium
Beryllium
Cadmium
Calcium
Chromium
Cobalt
Copper
Iron
Lead
Magnesium
Manganese
Molybdenum
Nickel
Potassium
Silver
Sodium
Thallium
Vanadium
Zinc

1.3 This digestion procedure is not suitable for samples which will be analyzed by graphite furnace atomic absorption spectroscopy because hydrochloric acid can cause interferences during furnace atomization.

2.0 SUMMARY OF METHOD

2.1 A mixture of HNO_3 and the material to be analyzed is refluxed in a covered Griffin beaker. This step is repeated with additional portions of HNO_3 until the digestate is light in color or until its color has stabilized. After the digestate has been brought to a low volume, it is refluxed with HCl and brought up to volume. If sample should go to dryness, it must be discarded and the sample reprepared.

3.0 INTERFERENCES

3.1 Interferences are discussed in the referring analytical method.

4.0 APPARATUS AND MATERIALS

4.1 Griffin beakers: 150 mL.

4.2 Watch glasses: ribbed and plain.

4.3 Qualitative filter paper or centrifugation equipment.

5.0 REAGENTS

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrated nitric acid, reagent grade (HNO_3): Acid should be analyzed to determine level of impurities. If method blank is <MDL, then acid can be used.

5.3 Concentrated hydrochloric acid, reagent grade (HCl): Acid should be analyzed to determine level of impurities. If method blank is <MDL, then acid can be used.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Aqueous wastewaters must be acidified to a pH of <2 with HNO_3.

6.4 Nonaqueous samples shall be refrigerated, when possible, and analyzed as soon as possible.

7.0 PROCEDURE

7.1 Transfer a 100-mL representative aliquot of the well-mixed sample to a 150-mL Griffin beaker and add 3 mL of concentrated HNO_3. Cover the beaker with a ribbed watch glass. Place the beaker on a hot plate and cautiously evaporate to a low volume (5 mL), making certain that the sample does not boil and that no portion of the bottom of the beaker is allowed to go dry. Cool the beaker and add another 3-mL portion of concentrated HNO_3. Re-cover the beaker with a nonribbed watch glass and return to the hot plate. Increase the temperature of the hot plate so that a gentle reflux action occurs.

NOTE: If a sample is allowed to go to dryness, low recoveries will result. Should this occur, discard the sample and reprepare.

7.2 Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing). Again, uncover the beaker or use a ribbed watch glass, and evaporate to a low volume (3 mL), not allowing any portion of the bottom of the beaker to go dry. Cool the beaker. Add a small quantity of 1:1 HCl (10 mL/100 mL of final solution) and warm the beaker for an additional 15 min to dissolve any precipitate or residue resulting from evaporation.

7.3 Wash down the beaker wall and watch glass with Type II water and, when necessary, filter or centrifuge the sample to remove silicates and other insoluble material that could clog the nebulizer. Filtration should be done only if there is concern that insoluble materials may clog the nebulizer. This additional step can cause sample contamination unless the filter and filtering apparatus are thoroughly cleaned and prerinsed with dilute HNO_3. Adjust to the final volume of 100 mL with Type II water. The sample is now ready for analysis.

8.0 QUALITY CONTROL

8.1 For each analytical batch of samples processed, blanks (Type II water reagents) should be carried throughout the entire sample preparation and analytical process. These blanks will be useful in determining if samples are being contaminated.

8.2 Duplicate samples should be processed on a routine basis. Duplicate samples will be used to determine precision. The sample load will dictate the frequency, but 20% is recommended.

8.3 Spiked samples or standard reference materials should be employed to determine accuracy. A spiked sample should be included with each group of samples processed and whenever a new sample matrix is being analyzed.

8.4 The method of standard addition shall be used for the analysis od all EP extracts.

9.0 METHOD PERFORMANCE

9.1 No data provided.

10.0 REFERENCES

10.1 None required.

APPENDIX B

RAW DATA

TABLE 14. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR ALUMINUM, AL IN REAGENT WATER

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		50.00	40.00	25.00	20.00	6.00	5.00	3.00	2.50
11		52.30	40.90	25.80	20.10	5.98	4.92	3.09	2.46
21	*	52.60*	43.30*	26.80*	21.20*	6.56*	5.24*	3.15*	2.82*
31		48.90	38.20	23.20	18.70	5.50	4.58	2.65	2.37
41	*	41.40*	25.10*	18.80*	17.30*	5.43*	3.61*	2.47*	1.41*
51		52.10	41.10	24.80	19.40	5.52	4.46	2.45	2.09
61		54.20	52.50	24.50	20.40	4.81	5.65	2.93	1.88
7		45.70	37.20	25.20	21.00	5.64	4.68	2.87	2.23
81		54.70	33.00	21.80	18.10	5.93	4.75	2.99	2.53
91		53.60	42.30	27.90	21.60	6.08	5.12	3.00	2.25

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR ALUMINUM, AL IN OHIO RIVER WATER

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		50.00	40.00	25.00	20.00	6.00	5.00	3.00	2.50
11		50.00	38.70	24.30	19.70	5.67	4.81	2.79	2.27
21		49.40	40.00	24.60	19.50	5.40	4.52	2.54	1.99
31		46.20	37.40	23.10	18.30	5.05	4.25	2.56	1.80
41	*	43.30*	22.00*	22.00*	16.70*	5.50*	4.37*	2.09*	1.52*
51		52.70	40.90	24.90	19.90	4.89	4.62	2.41	1.83
61		47.70	37.20	23.00	18.20	5.44	4.60	2.30	2.09
7		42.80	34.10	24.90	20.00	5.33	4.13	2.39	1.85
81		47.70	33.20	21.30	17.10	5.64	5.00	2.91	2.72
91	*	57.40*	42.80*	27.10*	21.40*	5.98*	4.82*	2.54*	2.12*

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

TABLE 14 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR ALUMINUM, AL IN MUNICIPAL SECONDARY EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		50.00	40.00	25.00	20.00	6.00	5.00	3.00	2.50
LAB/AN	LAB REJECTED								
11		50.40	39.20	24.80	20.10	5.85	4.82	2.80	2.45
21		50.40	40.20	25.20	20.00	6.01	5.13	2.82	2.38
31		48.30	39.40	23.80	18.80	5.54	4.28	2.37	2.39
41		34.60*	33.40	18.20	15.40	4.82	4.10	2.92	2.89
51		50.20	40.70	24.60	19.70	5.17	4.20	2.21	1.76
61	*	41.80*	31.00*	20.90*	16.50*	4.81*	4.18*	2.30*	2.09*
7		47.80	39.10	24.30	21.50	6.30	5.22	3.15	2.61
81		49.50	31.30	20.00	15.80	4.91	4.39	2.88	2.28
91	*	58.20*	46.90*	27.80*	22.90*	6.62*	5.31*	3.33*	2.05*

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR ALUMINUM, AL IN ELECTROPLATING EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		50.00	40.00	25.00	20.00	6.00	5.00	3.00	2.50
LAB/AN	LAB REJECTED								
11		48.90	38.30	24.70	19.60	5.79	4.89	2.93	2.50
21		51.40	42.00	26.00	20.70	6.23	5.13	3.37	2.82
31		50.40	40.70	24.80	20.20	5.69	4.60	2.73	1.98
41		44.00	38.00	20.90*	17.50	4.97	4.48	3.27	1.90
51		51.90	41.70	25.80	20.10	5.65	4.68	2.71	2.19
61		70.70*	55.00*	38.90*	26.80	7.95	3.56*	1.88	1.26
7		47.50	38.30	24.80	22.40	6.25	5.00	3.13	2.50
81	*	50.40*	33.50*	21.20*	17.00*	4.91*	4.19*	2.61*	2.30*
91	*	57.10*	45.20*	28.10*	22.70*	6.24*	5.43*	3.06*	2.53*

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 15. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR ANTIMONY, SB IN REAGENT WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		40.00	32.00	16.00	12.00	1.60	1.20	0.80	0.56
LAB/AN	LAB REJECTED								
11		45.80	35.10	16.80	12.20	1.44	1.00	0.73	0.41
21		44.10	34.60	17.20	12.60	1.49	1.18	0.63	0.36
32		40.10	32.00	15.70	12.20	1.49	1.23	0.73	0.61
41		48.80	40.20	20.60*	15.10*	2.54*	0.84	0.74	< 0.72*
51		44.50	33.20	16.50	12.40	1.24	0.90	0.56	0.39
62		44.00	47.20*	16.40	12.70	1.08	1.62	0.73	0.28
7		37.50	31.80	16.10	12.50	1.48	1.25	< 0.60*	< 0.60*
81		42.00	30.50	15.70	12.60	1.32	1.08	0.73	0.62
91		41.60	33.70	16.50	12.50	0.80	0.79	0.29*	0.09

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR ANTIMONY, SB IN OHIO RIVER WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		40.00	32.00	16.00	12.00	1.60	1.20	0.80	0.56
LAB/AN	LAB REJECTED								
11		44.80	34.50	16.40	12.30	1.67	1.19	0.87	0.55
21		42.80	34.10	17.10	11.90	1.36	1.00	0.63	0.36
32		39.50	32.40	15.60	11.40	1.39	1.05	0.70	0.48
41		51.80	39.20	19.30*	10.40	0.94	0.88	< 0.73*	< 0.73*
51	*	42.40*	34.40*	16.70*	12.60*	1.76*	1.35*	0.95*	0.77*
62		47.20	41.40	16.00	12.00	1.53	0.99	0.82	0.46
7		37.20	34.00	15.70	11.30	1.60	0.85	< 0.60*	< 0.60*
81		40.30	32.30	15.70	11.80	1.49	1.33	0.76	< 0.59*
91		42.60	34.00	16.60	12.30	1.29	1.01	0.91	0.57

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 15 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR ANTIMONY, SB IN MUNICIPAL SECONDARY EFFLUENT

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		40.00	32.00	16.00	12.00	1.60	1.20	0.80	0.56
11		41.50	33.30	16.90	12.80	1.67	1.35	1.07	0.66
21		43.40	33.90	16.80	12.20	1.36	0.95	0.59	0.36
32	*	36.30*	27.20*	14.30*	11.00*	1.23*	0.56*	0.71*	0.56*
41		43.40	37.00	18.50	14.90*	1.64	0.74	< 0.72*	< 0.72*
51	*	41.80*	33.90*	17.20*	12.90*	1.99*	1.57*	1.27*	1.08*
62		41.70	37.70	16.10	11.90	1.68	1.06	0.80	< 0.25*
7		42.00	35.20	15.50	11.70	1.59	1.25	0.68	< 0.60*
81		39.00	31.20	15.50	11.60	1.27	1.10	0.86	< 0.57*
91		42.10	32.90	16.10	12.20	1.47	1.18	0.60	0.16

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR ANTIMONY, SB IN ELECTROPLATING EFFLUENT

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		40.00	32.00	16.00	12.00	1.60	1.20	0.80	0.56
11		42.30	33.80	17.00	13.00	1.76	1.27	1.01	0.62
21		42.90	34.40	16.80	12.70	1.49	1.04	0.63	0.45
32		41.60	33.40	14.10*	10.40*	1.76	1.14	0.87	0.52
41	*	48.20*	36.90*	18.60*	15.90*	4.43*	1.64*	1.44*	1.64*
51		42.10	33.60	16.60	12.50	1.50	1.02	0.66	0.48
62		51.20*	39.60*	16.80	12.40	1.70	1.35	1.00	0.55
7		41.30	33.70	16.50	12.30	2.17	1.52	0.98	0.87*
81	*	40.70*	31.50*	15.40*	11.90*	1.42*	1.31*	0.84*	< 0.58*
91		41.70	33.40	16.60	12.50	1.84	1.39	0.93	0.61

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 16. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR BARIUM, BA IN REAGENT WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		20.00	16.00	8.00	6.00	1.20	1.00	0.60	0.52
LAB/AN	LAB REJECTED								
11		19.00	15.10	7.37	5.38	1.07	0.89	0.57	0.47
21		21.20	17.60	8.72	6.40	1.19	0.96	0.55	0.49
31		11.60	11.20	5.83	4.46	1.39	1.24	0.86*	0.62
41		15.30	15.00	7.27	5.64	1.25	0.94	0.47	0.54
51		20.80	17.10	8.67	6.66	1.26	1.04	0.63	0.50
61		16.00	15.50	7.89	6.71	1.23	0.84	0.54	0.42
7		19.30	15.30	8.43	5.87	1.22	1.02	0.63	0.51
81		19.90	14.70	7.65	6.34	1.02	0.87	0.63	0.56
91	*	22.70*	18.20*	8.57*	6.27*	1.36*	1.17*	0.69*	0.58*

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR BARIUM, BA IN OHIO RIVER WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		20.00	16.00	8.00	6.00	1.20	1.00	0.60	0.52
LAB/AN	LAB REJECTED								
11		19.40	15.20	7.40	5.38	1.11	0.98	0.50	0.49
21		20.40	16.50	9.84	6.64	1.25	1.13	0.73	0.61
31	*	19.40*	16.80*	12.10*	8.38*	2.10*	1.59*	1.05*	0.80*
41	*	13.90*	13.10*	5.48*	5.11*	1.09*	0.71*	0.44*	0.51*
51		20.60	17.30	8.62	6.50	1.29	1.16	0.67	0.61
61		8.00	10.90*	7.59	6.12	1.27	0.97	0.50	0.37
7		15.50	15.00	5.40	5.90	1.24	1.06	0.69	0.62
81		12.90	16.40	8.96	6.30	1.20	0.96	0.53	0.45
91		22.10	17.50	8.47	6.05	0.87*	0.63*	0.23	0.06

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 16 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR BARIUM, BA IN MUNICIPAL SECONDARY EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		20.00	16.00	8.00	6.00	1.20	1.00	0.60	0.52
LAB/AN	LAB REJECTED								
11		12.90	11.40	7.06	5.69	1.28	1.10	0.68	0.60
21		7.74	7.51	6.29	6.06	1.42	1.19	0.78	0.67
31		11.20	11.00	7.60	5.24	1.55	1.28	0.79	0.59
41	*	4.84*	3.99*	3.99*	3.65*	1.19*	0.92*	0.65*	0.37*
51		1.85	2.24	2.79*	4.67	1.30	1.11	0.68	0.57
61		3.72	8.05	6.04	4.80	1.67	1.19	0.73	0.84
7		3.49	3.40	5.69	4.86	1.26	1.09	0.74	0.63
81		9.66	9.38	5.81	5.35	1.41	1.26	0.66	0.64
91		13.10	8.85	7.36	6.24	1.04	0.78*	0.54	0.28

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR BARIUM, BA IN ELECTROPLATING EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		20.00	16.00	8.00	6.00	1.20	1.00	0.60	0.52
LAB/AN	LAB REJECTED								
11		12.70*	11.10	7.89	6.39	1.28	1.07	0.58	0.55
21		3.97	6.17	4.03	5.71	1.36	1.13	0.67	0.61
31	*	10.50*	6.43*	7.80*	5.68*	1.77*	1.42*	0.87*	0.58*
41		0.91	0.98	1.08	2.09	0.94	0.74	0.40	0.54
51		1.87	1.93	3.22	6.01	1.50	1.26	0.80	0.70
61	*	0.50*	0.70*	0.97*	1.82*	1.14*	0.71*	0.38*	0.26*
7		1.53	1.27	1.60	3.05	1.27	1.16	0.58	0.58
81		0.36	0.82	1.20	1.82	1.30	1.12	0.52	0.36
91		2.03	1.70	3.67	5.86	1.18	0.97	0.51	0.43

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 17. STUDY 38, SW 846 METHOD 3010, TRACE METALS, AA FLAME

RAW DATA FOR BERYLLIUM, BE IN REAGENT WATER

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		2.00	1.60	0.80	0.60	0.06	0.04	0.03	0.02
11		1.45	1.18	0.60	0.47	0.05	0.04	0.03	0.03
21	*	2.02*	1.61*	0.82*	0.62*	0.06*	0.04*	0.03*	0.02*
32		1.76	1.42	0.71	0.54	0.06	0.04	0.03	0.02
41		1.98	1.65	0.79	0.58	0.06	0.04	0.03	0.02
51	*	2.08*	1.62*	0.80*	0.61*	0.35*	0.32*	0.30*	0.02*
61		1.82	2.59*	0.77	0.57	0.04	0.04	0.02	< 0.00*
7		1.99	1.59	0.77	0.58	0.06	0.04	0.03	0.02
81		1.42	1.15	0.61	0.47	0.05	0.03*	0.03	0.02
91		1.86	1.48	0.71	0.52	0.02	0.01*	0.01	0.01

STUDY 38, SW 846 METHOD 3010, TRACE METALS, AA FLAME

RAW DATA FOR BERYLLIUM, BE IN OHIO RIVER WATER

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		2.00	1.60	0.80	0.60	0.06	0.04	0.03	0.02
11		1.37*	1.13*	0.50*	0.47	0.05	0.03	0.03	0.02
21		1.95	1.53	0.79	0.60	0.06	0.04	0.03	0.02
32		1.74	1.41	0.72	0.53	0.05	0.03	0.03	0.02
41		2.02	1.60	0.79	0.57	0.06	0.04	0.03	0.03
51	*	2.05*	1.64*	0.80*	0.60*	0.26*	0.24*	0.23*	0.02*
61		2.04	1.56	0.77	0.58	0.06	0.04	0.00	< 0.00*
7		1.94	1.55	0.78	0.58	0.06	0.04	0.03	0.02
81	*	1.49*	1.22*	0.62*	0.48*	0.05*	0.03*	0.03*	0.02*
91		1.83	1.53	0.70	0.53	0.03	0.01*	0.01	0.01

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 17 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR BERYLLIUM, BE IN MUNICIPAL SECONDARY EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		2.00	1.60	0.80	0.60	0.06	0.04	0.03	0.02
LAB/AN	LAB REJECTED								
11		2.03	1.61	0.80	0.60	0.06	0.04	0.04	0.03
21		1.99	1.54	0.79	0.59	0.06	0.04	0.03	0.02
32		1.75	1.41	0.70	0.53	0.06	0.04	0.03	0.02
41		2.00	1.54	0.84	0.56	0.06	0.03	0.03	0.02
51		2.00	1.61	0.80	0.61	0.09*	0.06*	0.02	0.01
61	*	2.16*	1.64*	0.76*	0.58*	0.08*	0.05*	0.04*	0.04*
7		1.90	1.55	0.78	0.57	0.06	0.04	0.03	0.02
81	*	1.46*	1.21*	0.62*	0.48*	0.05*	0.03*	0.03*	0.01*
91	*	1.85*	1.51*	0.70*	0.52*	0.04*	0.01*	0.02*	0.02*

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR BERYLLIUM, BE IN ELECTROPLATING EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		2.00	1.60	0.80	0.60	0.06	0.04	0.03	0.02
LAB/AN	LAB REJECTED								
11	*	2.05*	1.64*	0.82*	0.61*	0.06*	0.04*	0.03*	0.02*
21		1.99	1.60	0.80	0.61	0.06	0.04	0.03	0.02
32		1.78*	1.45	0.72	0.55	0.05	0.03*	0.03	0.02
41		1.96	1.48	0.74	0.57	0.05	0.04	0.03	0.02
51	*	2.02*	1.59*	0.80*	0.58*	0.09*	0.08*	0.07*	0.02*
61		2.02	1.57	0.72	0.57	0.06	0.04	0.02	0.02
7		1.97	1.60	0.78	0.59	0.06	0.04*	0.03	0.03
81	*	1.47*	1.22*	0.61*	0.47*	0.04*	0.02*	0.01*	0.01*
91	*	1.79*	1.48*	0.71*	0.51*	0.03*	0.02*	0.02*	0.01*

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 18. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR CADMIUM, CD IN REAGENT WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		2.00	1.60	0.80	0.60	0.06	0.04	0.03	0.02
LAB/AN	LAB REJECTED								
11		1.92	1.52	0.77	0.57	0.06	0.04	0.03	0.03
21		1.99	1.59	0.79	0.59	0.07	0.05	0.04	0.03
31		1.97	1.55	0.76	0.57	0.06	0.04	0.03	0.02
41		2.02	1.62	0.78	0.61	0.06	0.04	< 0.03*	< 0.03*
51		2.02	1.63	0.81	0.58	0.06	0.04	0.03	0.02
63		1.70*	2.28*	0.86	0.68*	0.03*	0.02*	0.01*	< 0.01*
7		1.97	1.62	0.78	0.57	0.06	0.04	0.04	0.03
81		2.05	1.54	0.82	0.61	0.06	0.04	0.03	0.02
91		2.04	1.68	0.82	0.62	0.06	0.04	0.03	0.03

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR CADMIUM, CD IN OHIO RIVER WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		2.00	1.60	0.80	0.60	0.06	0.04	0.03	0.02
LAB/AN	LAB REJECTED								
11		1.39	1.50	0.74	0.56	0.06	0.04	0.03	0.02
21		1.96	1.54	0.77	0.57	0.06	0.04	0.03	0.01*
31		1.94	1.53	0.76	0.56	0.05	0.04	0.03	0.02
41		1.98	1.57	0.80	0.59	0.06	0.04	< 0.03*	< 0.03*
51		1.99	1.57	0.80	0.58	0.05	0.03	0.03	0.02
63		2.04	1.60	0.82	0.64	0.05	0.04	0.04	0.02
7		1.94	1.62	0.80	0.59	0.06	0.04	0.04	< 0.03*
81		2.08	1.60	0.82	0.62	0.06	0.04	0.03	0.02
91	*	2.07*	1.68*	0.83*	0.63*	0.07*	0.04*	0.03*	0.03*

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 18 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR CADMIUM, CD IN MUNICIPAL SECONDARY EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		2.00	1.60	0.80	0.60	0.06	0.04	0.03	0.02
LAB/AN	LAB REJECTED								
11	*	1.92*	1.52*	0.77*	0.55*	0.05*	0.03*	0.02*	0.01*
21		1.98	1.53	0.76	0.56	0.05	0.04	0.03	0.02
31		2.00	1.53	0.76	0.57	0.06	0.04	0.03	0.02
41	*	2.10*	1.54*	0.77*	0.61*	0.08*	0.05*	0.04*	0.04*
51		2.05	1.59	0.82	0.60	0.06	0.04	0.03	0.02
63		2.12	1.63	0.72	0.54	0.04	0.02*	< 0.01*	< 0.01*
7		1.93	1.63	0.80	0.58	0.07	0.04	0.03	0.03
81		1.98	1.58	0.81	0.62	0.06	0.04	0.03	0.02
91		2.08	1.67	0.80	0.60	0.06	0.04	0.03	0.02

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR CADMIUM, CD IN ELECTROPLATING EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		2.00	1.60	0.80	0.60	0.06	0.04	0.03	0.02
LAB/AN	LAB REJECTED								
11		1.96	1.56	0.77	0.59	0.06	0.04	0.04	0.03
21		1.95	1.58	0.77	0.58	0.06	0.04	0.03	0.02
31	*	1.97*	1.51*	0.75*	0.57*	0.05*	0.03*	0.03*	0.02*
41		2.02	1.55	0.77	0.59	0.07	0.07*	< 0.04*	< 0.04*
51		2.01	1.64	0.85	0.61	0.06	0.04	0.03	0.02
63	*	1.99*	1.56*	0.87*	0.67*	0.08*	0.08*	0.05*	0.05*
7		1.92	1.63	0.78	0.58	0.07	0.05	0.03	< 0.03*
81		2.03	1.59	0.80	0.62	0.06	0.04	0.04	0.02
91		2.05	1.68	0.81	0.59	0.05	0.03	0.03	0.02

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 19. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR CALCIUM, CA IN REAGENT WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		120.00	90.00	60.00	45.00	24.00	18.00	12.00	9.00
LAB/AN	LAB REJECTED								
11		117.00	86.40	60.60	44.70	24.60	18.70	12.50	9.30
21		110.00	63.20	52.80	63.10	27.20	24.70	15.20	10.10
31		116.00	88.10	60.70	45.10	22.80	16.10	12.10	8.90
41		85.30	68.20	40.50	33.20	22.70	17.10	12.50	9.42
51		117.00	86.60	57.40	43.40	24.60	18.40	11.30	8.49
62		140.00	122.00	63.00	50.40	16.40*	23.20	16.70	7.59
7		157.00	111.00	73.90	54.90	28.60	20.60	10.80	8.09
81		129.00	97.40	65.20	52.80	26.60	21.70	12.30	9.23
91		124.00	97.90	64.30	47.90	26.20	19.50	12.90	9.89

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR CALCIUM, CA IN OHIO RIVER WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		120.30	90.30	60.00	45.00	24.00	18.00	12.00	9.00
LAB/AN	LAB REJECTED								
11		113.00	79.60	52.50	41.00	21.30	16.50	11.10	8.70
21	*	62.00*	44.50*	35.10*	22.00*	13.40*	8.62*	7.64*	2.94*
31		116.30	88.10	57.60	45.40	18.40	15.00	7.20	3.10
41		94.90	73.90	40.30	29.00	21.20	15.50	10.70	7.30
51	*	104.00*	73.40*	41.70*	31.50*	10.00*	4.55*	< 0.01*	< 0.01*
62		91.00	88.00	57.20	42.80	21.90	15.40	11.80	7.16
7		147.00	106.00	63.50	33.30	23.80	15.20	8.04	2.35
81	*	126.00*	94.70*	63.80*	46.00*	24.80*	19.00*	14.30*	11.10*
91		126.00	92.00	63.40	47.20	21.60	16.30	7.75	5.05

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 19 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR CALCIUM, CA IN MUNICIPAL SECONDARY EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		120.00	90.00	60.00	45.00	24.00	18.00	12.00	9.00
LAB/AN	LAB REJECTED								
11		106.00	82.00	54.10	43.10	22.90	18.50	12.40	9.40
21	*	80.90*	50.30*	33.20*	40.40*	23.90*	14.90*	1.80*	3.46*
31		116.00	87.30	56.40	43.60	24.60	15.90	10.20	9.20
41		125.00	102.00	58.30	50.60	36.30*	21.90	17.60	16.00*
51	*	101.00*	67.60*	39.70*	26.10*	4.72*	0.52*	< 0.01*	< 0.01*
62	*	115.00*	90.40*	93.90*	77.00*	41.60*	52.60*	47.10*	52.60*
7		137.00	117.00	72.20	56.30	24.90	17.50	11.80	7.71
81		120.00	89.60	58.80	45.80	23.90	20.70	16.20	8.90
91		127.00	97.20	74.00	47.60	24.20	16.50	12.00	7.70

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR CALCIUM, CA IN ELECTROPLATING EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		120.00	90.00	60.00	45.00	24.00	18.00	12.00	9.00
LAB/AN	LAB REJECTED								
11		117.00	81.20	54.90	41.20	21.10	17.40	9.90	7.40
21		78.00	56.70	46.30	37.30	19.30	23.60	18.00	39.50*
31		112.00	84.70	56.70	42.50	22.70	15.80	10.00	7.10
41		100.00	81.00	43.10	33.40	24.00	19.70	14.10	10.10
51		111.00	88.60	58.40	43.40	23.10	17.60	12.80	6.74
62	*	96.00*	65.20*	40.30*	23.40*	7.99*	1.24*	< 0.01*	< 0.01*
7	*	162.00*	118.00*	76.80*	56.00*	30.50*	27.00*	14.80*	11.00*
81		130.00	89.30	64.40	45.90	27.80	18.20	10.80	9.10
91		135.00	99.70	66.10	44.10	23.90	19.60	9.75	8.00

* = REJECTED

CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 20. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR CHROMIUM, CR IN REAGENT WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		10.00	8.00	5.00	4.00	0.60	0.50	0.30	0.24
LAB/AN	LAB REJECTED								
11	*	8.68*	7.26*	4.52*	3.72*	0.55*	0.48*	0.28*	0.22*
21		10.50	8.36	5.21	4.23	0.66	0.54	0.30	0.26
31		12.30	9.49	5.76	4.62	0.63	0.49	0.27	0.18
41		11.00	8.12	5.20	4.12	0.54	0.43	0.24	0.20
51		12.00	9.14	5.59	4.62	0.59	0.51	0.30	0.23
61		12.00	13.80*	5.71	4.87	0.49	0.64	0.29	0.26
7		10.20	8.29	4.87	4.05	0.63	0.54	0.34	0.27
81		11.70	8.70	5.61	4.53	0.66	0.53	0.37	0.27
91		8.99	7.13	4.55	3.67	0.65	0.51	0.33	0.26

STUDY 38,SW 946 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR CHROMIUM, CR IN OHIO RIVER WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		10.00	8.00	5.00	4.00	0.60	0.50	0.30	0.24
LAB/AN	LAB REJECTED								
11	*	8.47*	6.71*	4.17*	3.26*	0.57*	0.49*	0.30*	0.22*
21		10.10	7.32	5.04	4.05	0.58	0.49	0.27	0.24
31		12.13	9.30	5.73	4.33	0.69	0.55	0.34	0.29
41	*	12.30*	10.10*	5.77*	4.27*	0.73*	0.69*	0.37*	0.32*
51	*	12.20*	9.22*	5.65*	4.46*	0.75*	0.68*	0.42*	0.33*
61		11.40	8.93	5.64	4.61	0.70	0.61	0.32	0.17
7		9.97	8.18	4.54	3.64	0.59	0.46	0.28	0.23
81		12.20	9.19	5.69	4.61	0.70	0.60	0.32	0.21
91	*	9.00*	7.28*	4.45*	3.65*	0.41*	0.38*	0.27*	0.21*

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 20 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR CHROMIUM, CR IN MUNICIPAL SECONDARY EFFLUENT

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		10.00	8.00	5.00	4.00	0.60	0.50	0.30	0.24
11	*	8.11*	6.61*	4.41*	3.52*	0.39*	0.40*	< 0.01*	< 0.01*
21		10.20	7.78	4.97	3.90	0.58	0.50	0.26	0.19
31		12.10	9.24	5.90	4.58	0.54	0.44	0.33	0.28
41		12.20	9.06	5.38	4.12	0.64	0.43	0.39	0.37
51		12.10	9.92	5.67	4.58	0.70	0.63	0.38	0.30
61	*	13.00*	11.50*	18.00*	5.77*	1.83*	1.77*	1.56*	1.62*
7		10.50	8.46	5.39	3.80	0.56	0.48	0.27	0.24
81		11.30	9.51	5.63	4.51	0.56	0.56	0.30	0.26
91	*	8.98*	7.13*	4.27*	3.51*	0.40*	0.36*	0.23*	0.06*

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR CHROMIUM, CR IN ELECTROPLATING EFFLUENT

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		10.00	8.00	5.00	4.00	0.60	0.50	0.30	0.24
11		8.87	7.10	4.62	3.74	0.73	0.65	0.45	0.40
21		10.20	8.24	5.09	4.11	0.62	0.53	0.32	0.26
31		12.40	9.78	5.89	4.57	0.66	0.49	0.29	0.25
41		11.70	8.86	5.42	4.18	0.64	0.55	0.36	0.34
51	*	12.40*	9.37*	5.81*	4.79*	0.79*	0.67*	0.41*	0.31*
61		12.30	9.15	5.51	4.29	0.20*	0.23	0.03	< 0.02*
7		9.83	8.38	4.67	3.79	0.52	0.42	0.32	0.25
81		11.70	9.11	5.79	4.65	0.79	0.66	0.46	0.34
91	*	9.01*	7.22*	4.58*	3.57*	0.56*	0.43*	0.26*	0.23*

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 21. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR COBALT, CO IN REAGENT WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		15.00	12.50	7.50	6.00	3.50	2.50	1.50	1.00
LAB/AN	LAB REJECTED								
11		13.90	11.70	7.32	5.76	3.37	2.42	1.48	0.97
21	*	17.00*	13.80*	8.81*	6.79*	3.85*	2.63*	1.56*	1.03*
32		14.90	12.10	7.22	5.83	3.41	2.36	1.44	0.94
41	*	14.40*	11.70*	6.88*	5.30*	3.05*	2.20*	1.45*	0.74*
51		15.50	12.70	7.80	6.26	3.60	2.55	1.47	0.92
62		16.60	10.30*	7.02	6.00	2.54*	3.52*	1.52	1.01
7		14.20	11.70	7.11	5.77	3.30	2.37	1.48	1.00
81	*	16.30*	13.10*	7.94*	6.35*	3.65*	2.58*	1.60*	1.03*
91		13.60	12.10	6.82	5.55	3.50	2.57	1.60	1.06

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR COBALT, CO IN OHIO RIVER WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		15.00	12.50	7.50	6.00	3.50	2.50	1.50	1.00
LAB/AN	LAB REJECTED								
11		13.60	11.30	6.92	5.71	3.29	2.41	1.45	1.00
21	*	16.70*	13.90*	7.90*	7.15*	3.88*	2.76*	1.67*	1.15*
32		14.00	11.70	7.28	5.74	3.29	2.37	1.47	0.96
41	*	13.40*	11.70*	6.52*	5.43*	2.73*	2.00*	1.36*	0.82*
51		15.40	12.90	7.99	6.42	3.64	2.72	1.64	1.09
62		16.30	14.60	5.96	5.07	2.98	2.09	1.19	0.76
7		14.30	11.90	7.32	6.12	3.52	2.26	1.51	0.98
81		15.90	13.60	8.08	6.33	3.68	2.66	1.57	1.09
91		14.10	11.50	6.90	5.59	3.17	2.30	1.44	0.98

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 21 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR COBALT, CO IN MUNICIPAL SECONDARY EFFLUENT

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		15.00	12.50	7.50	6.00	3.50	2.50	1.50	1.00
11	*	11.30*	9.78*	6.27*	5.13*	2.97*	2.16*	1.26*	0.94*
21		18.30*	14.60	8.15	7.37	4.39	3.21	1.65	1.17
32		14.30	11.50	6.80	5.60	3.70	2.30	1.40	0.80
41		14.30	12.80	7.52	5.42	3.62	3.02	1.62	1.42
51		15.30	12.90	7.48	6.16	3.45	2.93	1.81	1.43
62	*	27.00*	23.60*	17.20*	15.70*	13.90*	13.30*	9.62*	9.79*
7		13.60	11.30	7.29	5.56	3.39	2.64	1.51	0.94
81		15.30	13.80	8.39	6.71	4.05	3.14	2.06	1.73
91		14.80	12.00	7.74	5.86	3.70	2.33	1.50	0.65

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR COBALT, CO IN ELECTROPLATING EFFLUENT

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		15.00	12.50	7.50	6.00	3.50	2.50	1.50	1.00
11		14.10	11.90	7.31	5.91	3.42	2.52	1.53	1.01
21	*	16.90*	13.30*	8.63*	6.79*	3.73*	2.60*	1.53*	1.05*
32		14.90	12.20	7.09	5.72	3.30	2.31	1.39	0.91
41	*	13.70*	11.70*	6.97*	5.66*	3.00*	2.25*	1.29*	0.89*
51	*	15.30*	13.00*	7.92*	6.35*	3.75*	2.64*	1.58*	0.99*
62		17.60	17.20*	7.27	5.78	3.48	2.63	1.60	0.97
7		14.00	12.00	7.27	5.82	3.27	2.36	1.46	0.95
81		15.90	12.10	7.88*	6.37*	3.53	2.51	1.50	0.98
91	*	14.10*	12.20*	6.92*	5.34*	2.98*	2.12*	1.31*	0.90*

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 22. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR COPPER , CU IN REAGENT WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		5.00	4.00	2.00	1.50	0.25	0.20	0.15	0.10
LAB/AN	LAB REJECTED								
11		4.86	3.82	1.94	1.44	0.24	0.19	0.15	0.10
21		4.90	3.99	2.04	1.50	0.26	0.22	0.15	0.10
31	*	4.67*	3.69*	1.87*	1.40*	0.21*	0.17*	0.12*	0.07*
41		5.17	3.96	2.08	1.54	0.26	0.18	0.13*	0.10
51		4.95	3.96	1.96	1.46	0.26	0.21	0.15	0.11
62	*	5.22*	5.38*	2.18*	1.58*	0.27*	0.21*	0.15*	0.11*
7		4.95	3.79	1.91	1.45	0.26	0.20	0.15	0.12
81		5.15	3.97	2.03	1.51	0.24	0.21	0.15	0.11
91		5.19	4.27	2.06	1.56	0.27	0.21	0.15	0.10

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR COPPER , CU IN OHIO RIVER WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		5.00	4.00	2.00	1.50	0.25	0.20	0.15	0.10
LAB/AN	LAB REJECTED								
11	*	4.79*	3.79*	1.88*	1.43*	0.23*	0.19*	0.14*	0.09*
21		4.71	3.81	1.93	1.43	0.24	0.19	0.15	0.09*
31		4.97	3.34	1.99	1.37	0.24	0.19	0.15	0.10
41		5.03	4.03	2.01	1.50	0.26	0.21	0.15	0.10
51		4.90	3.88	1.97	1.47	0.25	0.19	0.14	0.10
62		4.85	4.10	2.16*	1.58	0.23	0.18	0.14	0.10
7		4.97	3.86	1.97	1.50	0.29	0.24	0.19*	0.14*
81		5.24	4.09	2.01	1.55	0.25	0.20	0.15	0.10
91	*	5.39*	4.35*	2.10*	1.58*	0.27*	0.21*	0.16*	0.11*

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 22 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR COPPER , CU IN MUNICIPAL SECONDARY EFFLUENT

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		5.00	4.00	2.00	1.50	0.25	0.20	0.15	0.10
11		4.78	3.89	2.06	1.57	0.27	0.22	0.16	0.11
21	*	4.69*	3.61*	1.90*	1.44*	0.23*	0.19*	0.14*	0.09*
31		4.97	3.95	1.95	1.49	0.25	0.19	0.14	0.09
41		5.08	4.02	1.99	1.56	0.27	0.22	0.17	0.12
51		4.71	3.85	1.93	1.47	0.24	0.19	0.15	0.09
62		5.06	4.19	2.00	1.48	0.16*	0.12*	0.05*	< 0.02*
7		4.86	3.95	1.96	1.49	0.26	0.19	0.14	0.10
81		5.01	4.06	2.04	1.51	0.25	0.21	0.16	0.11
91		5.39	4.20	2.08	1.58	0.26	0.20	0.16	0.06

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR COPPER , CU IN ELECTROPLATING EFFLUENT

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		5.00	4.00	2.00	1.50	0.25	0.20	0.15	0.10
11		4.84	3.94	2.09	1.57	0.27	0.23	0.17	0.13
21		4.75	3.05	1.72	1.48	0.25	0.20	0.14	0.10
31		4.71	3.79	1.94	1.48	0.24	0.19	0.15	0.10
41		4.82	4.00	1.97	1.53	0.24	0.19	0.16	0.10
51	*	4.80*	3.90*	1.89*	1.41*	0.23*	0.19*	0.14*	0.09*
62	*	5.26*	4.37*	2.26*	1.69*	0.29*	0.25*	0.19*	0.14*
7		4.83	3.96	1.94	1.49	0.24	0.19	0.14	0.09
81		5.20	4.10	2.03	1.54	0.25	0.20	0.15	0.10
91		5.31	4.39	2.06	1.51	0.25	0.21	0.16	0.11

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 23. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR IRON, FE IN REAGENT WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		5.00	4.00	2.50	2.00	0.40	0.30	0.20	0.15
LAB/AN	LAB REJECTED								
11		4.83	3.82	2.44	1.92	0.39	0.29	0.20	0.13
21		5.19	3.99	2.55	2.08	0.43	0.32	0.22	0.16
31		4.61	3.65	2.28	1.82	0.35	0.26	0.18	0.13
41	*	5.57*	4.26*	2.58*	2.20*	0.46*	0.46*	0.24*	0.17*
51		4.54	3.81	2.40	1.93	0.37	0.28	0.17	0.12
62		4.68	5.73*	2.61	1.94	0.28	0.38	0.21	0.07*
7		4.62	3.50	2.16	1.74	0.37	0.28	0.18	0.14
81	*	5.18*	4.01*	2.59*	2.07*	0.44*	0.32*	0.22*	0.21*
91		3.72	3.14	1.97	1.68	0.40	0.26	0.18	0.14

STUDY 38,SW 946 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR IRON, FE IN OHIO RIVER WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		5.00	4.00	2.50	2.00	0.40	0.30	0.20	0.15
LAB/AN	LAB REJECTED								
11		4.80	3.80	2.30	1.93	0.35	0.27	0.18	0.13
21		4.79	3.33	2.37	1.92	0.31	0.24	0.15	0.11
31		4.42	3.50	2.24	1.68	0.21	0.15	0.10	0.05
41		5.47	4.68	2.47	1.97	0.18	0.35	< 0.16*	< 0.16*
51		4.87	3.94	2.15	1.72	0.16	0.07	0.03	< 0.01*
62		4.34	3.03	1.95	2.05	0.03	0.03	0.21	< 0.02*
7		4.48	3.64	2.25	1.66	0.33	0.21	0.16	0.10
81	*	5.20*	4.07*	2.48*	2.05*	0.33*	0.26*	0.17*	0.13*
91	*	3.72*	2.86*	1.91*	1.54*	0.09*	0.08*	0.03*	0.03*

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 23 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR IRON, FE IN MUNICIPAL SECONDARY EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		5.00	4.00	2.50	2.00	0.40	0.30	0.20	0.15
LAB/AN	LAB REJECTED								
11		4.88	3.80	2.41	1.94	0.40	0.34	0.18	0.20*
21		4.89	3.85	2.44	2.01	0.40	0.32	0.21	0.11
31		4.62	3.79	2.34	1.94	0.28	0.16	0.14	0.11
41		3.13*	1.24*	2.58	2.20	0.59	0.23	0.23	0.34*
51		4.97	3.64	2.17	1.70	0.16	0.08	< 0.01*	< 0.01*
62	*	7.62*	5.87*	3.55*	3.25*	1.60*	1.50*	1.57*	1.67*
7		4.70	3.63	2.32	1.89	0.37	0.25	0.20	0.13
81		5.04	4.10	2.60	2.06	0.35	0.29	0.25	0.11
91	*	3.85*	3.06*	2.08*	1.68*	0.07*	0.04*	0.03*	0.11*

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR IRON, FE IN ELECTROPLATING EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		5.00	4.00	2.50	2.00	0.40	0.30	0.20	0.15
LAB/AN	LAB REJECTED								
11		4.86	3.73	2.37	1.91	0.28	0.29	0.17	0.10
21		4.30	3.92	2.34	1.88	0.27	0.23	0.12	0.02
31	*	4.74*	3.78*	2.33*	1.85*	0.21*	0.10*	0.07*	0.07*
41		5.26	4.44	2.52	2.14	0.60	0.56*	0.51	0.32
51		5.67	4.76	2.59	1.99	0.38	0.22	0.22	0.10
62		5.75	5.43	2.95	2.25	0.10	< 0.02*	< 0.02*	< 0.02*
7		4.72	3.30	2.39	2.08	0.16	0.13	0.10	0.08
81		5.30	4.16	2.55	2.16	0.19	0.13	0.06	0.08
91		4.47	3.79	2.69	2.09	0.27	0.27	0.28	0.40

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 24. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR LEAD, PB IN REAGENT WATER

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		20.00	16.00	8.00	6.00	1.20	0.80	0.60	0.40
11		20.50	16.30	8.39	6.23	1.26	0.87	0.65	0.43
21		20.60	16.10	8.10	6.14	1.33	0.92	0.70	0.52
31	*	18.40*	15.20*	7.99*	5.87*	1.12*	0.70*	0.50*	0.34*
41		21.20	15.30	7.23*	6.20	1.18	0.63	0.57	< 0.36*
51		20.80	16.50	8.16	6.12	1.23	0.84	0.58	0.42
62		24.70*	25.70*	11.80*	8.48*	1.00	1.29*	0.43	0.38
7		20.00	16.40	8.01	6.01	1.15	0.73	0.58	< 0.45*
81		20.20	16.30	8.35	6.22	1.13	0.77	0.54	0.32
91		20.90	17.50	8.60	6.41	1.35	0.86	0.68	0.41

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR LEAD, PB IN OHIO RIVER WATER

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		20.00	16.00	8.00	6.00	1.20	0.80	0.60	0.40
11		20.30	16.10	8.10	6.18	1.24	0.86	0.62	0.45
21		20.20	15.80	7.88	5.78	1.19	0.84	0.70	0.57
31		18.30	15.10	7.95	6.02	1.20	0.83	0.68	0.50
41		20.10	17.10	7.44	6.05	1.42	0.77	0.61	< 0.25*
51		20.80	16.40	8.62	6.48	1.31	0.90	0.68	0.50
62	*	24.80*	18.60*	12.40*	9.97*	1.34*	0.86*	0.72*	0.53*
7		19.60	15.70	9.03	6.30	1.24	0.88	0.60	< 0.45*
81		18.60	17.00	8.37	6.36	1.26	0.88	0.64	0.49
91		21.90	17.50	8.60	6.62	1.41	0.83	0.65	0.42

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS:
1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 24 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR LEAD, PB IN MUNICIPAL SECONDARY EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		20.00	16.00	8.00	6.00	1.20	0.80	0.60	0.40
LAB/AN	LAB REJECTED								
11		20.40	16.30	8.29	6.20	1.24	0.73	0.51	0.38
21		20.70	16.00	7.79	5.78	1.28	0.84	0.66	0.48
31	*	18.80*	15.40*	7.88*	5.86*	1.06*	0.65*	0.48*	0.30*
41		21.10	17.70	8.86	6.05	0.98	0.53	0.36	0.67
51		20.30	16.20	8.13	6.14	1.23	0.82	0.59	0.39
62		22.00	19.60	11.00*	6.72	1.15	0.62	0.52	0.48
7		18.50	15.90	7.96	5.89	1.19	0.77	0.62	< 0.45*
81		20.40	17.00	8.38	6.18	1.24	0.84	0.63	0.42
91		21.60	17.30	8.47	6.35	1.40	0.85	0.63	0.28

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR LEAD, PB IN ELECTROPLATING EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		20.00	16.00	8.30	6.00	1.20	0.80	0.60	0.40
LAB/AN	LAB REJECTED								
11									
21	*	18.20*	14.50*	7.00*	5.22*	1.26*	0.76*	0.58*	0.36*
31		18.30	15.10	7.99	5.70	1.26	0.78	0.58	0.45
41		16.70	16.30	7.23	5.85	0.90*	0.83	0.36*	0.51
51		19.90	15.60	8.09	6.05	1.21	0.78	0.62	0.41
62		22.70	18.80*	7.63	4.98	1.21	0.78	0.54	0.40
7		17.90	15.70	8.03	6.06	1.31	0.96*	0.62	0.50
81		18.70	16.70	8.25	6.35	1.23	0.83	0.64	0.41
91	*	21.40*	17.80*	8.67*	6.30*	1.39*	0.93*	0.72*	0.43*

* = REJECTED

CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 25. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR MAGNESIUM, MG IN REAGENT WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		40.00	30.00	20.00	15.00	7.00	5.00	3.00	2.00
LAB/AN	LAB REJECTED								
11		39.90	30.10	20.50	15.00	7.00	5.10	3.10	2.00
21		39.10	28.20	19.20	15.40	7.05	5.18	3.09	2.03
32	*	40.50*	29.90*	19.30*	14.50*	6.60*	4.60*	2.70*	1.70*
41		42.20	30.10	19.70	14.90	7.03	5.18	3.19	2.00
51		40.60	30.20	19.50	14.60	7.23	5.20	3.03	2.03
62		49.90*	51.00*	23.10	19.70*	5.73*	8.06*	3.44	1.92
7		44.50	32.60	20.50	15.80	7.33	5.08	2.91	1.99
81		41.70	30.90	21.20	16.70	7.79*	5.69*	3.01	2.04
91		40.90	31.50	21.10	15.60	7.09	5.08	3.18	2.08

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR MAGNESIUM, MG IN OHIO RIVER WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		40.00	30.00	20.00	15.00	7.00	5.00	3.00	2.00
LAB/AN	LAB REJECTED								
11		39.20	28.90	19.30	14.80	6.60	5.00	2.90	2.00
21	*	35.10*	26.00*	18.20*	13.70*	6.44*	4.54*	2.70*	2.01*
32		40.00	30.60	20.00	15.30	6.50	4.60	3.00	2.10
41		40.30	30.60	20.40	16.40	7.12	5.32	2.82	2.22
51		41.20	30.20	18.70	14.80	6.56	4.83	2.80	1.78
62	*	44.10*	37.90*	23.60*	19.20*	7.69*	6.96*	3.90*	0.71*
7		43.00	31.20	20.50	16.00	7.19	4.40	2.99	2.15
81	*	40.90*	31.40*	21.00*	15.80*	7.80*	5.80*	3.90*	2.90*
91		40.00	28.90	20.30	15.70	6.91	5.06	3.02	2.06

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 25 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR MAGNESIUM, MG IN MUNICIPAL SECONDARY EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		40.00	30.00	20.00	15.00	7.00	5.00	3.00	2.00
LAB/AN	LAB REJECTED								
11		38.20	28.60	18.80	15.40	7.30	5.40	3.30	2.40
21	*	34.50*	25.70*	18.00*	13.40*	6.27*	3.73*	2.81*	1.68*
32		46.50	30.30	19.60	15.10	7.20	4.90	3.20	2.20
41		41.10	32.30	19.40	18.20*	7.70	5.00	3.30	2.00
51		39.40	30.20	20.10	14.90	6.65	5.07	3.23	2.50
62	*	58.80*	46.90*	35.60*	32.00*	15.00*	23.40*	14.60*	19.90*
7		40.40	32.90	21.70	14.60	7.20	5.17	3.19	1.55
81	*	39.20*	28.50*	18.70*	14.50*	6.10*	4.70*	2.60*	1.00*
91		42.00	32.40	23.20	14.30	6.39	4.33	3.02	0.87

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR MAGNESIUM, MG IN ELECTROPLATING EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		40.00	30.00	20.00	15.00	7.00	5.00	3.00	2.00
LAB/AN	LAB REJECTED								
11		39.30	29.20	19.80	14.90	6.80	5.30	3.10	2.20
21	*	36.00*	27.40*	18.30*	13.30*	6.59*	4.65*	2.72*	1.94*
32		41.10	30.70	20.10	15.20	7.00	4.90	2.90	1.90
41		39.70	27.50	19.80	15.40	7.04	5.04	2.87	1.81
51		39.30	29.50	19.50	14.70	6.72	4.78	3.01	1.88
62	*	43.10*	42.70*	26.90*	23.70*	9.41*	11.10*	8.37*	7.80*
7		41.30	31.50	20.80	16.10	7.11	4.87	3.12	2.23
81		40.90	31.30	20.40	14.90	7.20	5.20	2.50	2.00
91		45.10*	33.40	23.10*	15.20	7.22	5.20	2.72	1.85

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 26. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR MANGANESE, MN IN REAGENT WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		3.00	2.50	1.20	1.00	0.15	0.10	0.07	0.05
LAB/AN	LAB REJECTED								
11		3.09	2.54	1.25	1.02	0.15	0.11	0.08	0.05
21		3.13	2.55	1.26	1.06	0.15	0.08	< 0.06*	< 0.06*
31	*	2.79*	2.29*	1.10*	0.91*	0.13*	0.09*	0.06*	0.04*
41		3.06	2.46	1.37	1.08	0.16	0.13	0.06	0.04
51		3.10	2.48	1.21	1.02	0.15	0.10	0.07	0.03
62		3.06	3.55*	1.30	1.18	0.04*	0.14	0.04	0.04
7		2.85*	2.34	1.12	0.96	0.15	0.10	0.07	0.05
81		3.13	2.52	1.25	1.03	0.17	0.12	0.09	0.07
91		3.02	2.58	1.17	0.99	0.14	0.10	0.06	0.04

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR MANGANESE, MN IN OHIO RIVER WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		3.00	2.50	1.20	1.00	0.15	0.10	0.07	0.05
LAB/AN	LAB REJECTED								
11		3.04	2.52	1.20	1.02	0.15	0.10	0.06	0.05
21		3.03	2.48	1.16	0.96	0.13	0.07	< 0.06*	< 0.06*
31		2.82	2.30	1.12	0.88	0.12	0.08	0.05	0.03
41	*	3.23*	2.84*	1.30*	1.10*	0.18*	0.12*	0.07*	0.06*
51		3.21	2.57	1.17	0.98	0.12	0.07	0.04	0.02
62		15.30*	12.40*	1.32*	1.04	0.17	0.09	< 0.05*	< 0.05*
7		2.84	2.27	1.12	0.94	0.13	0.08	0.05	0.02
81	*	3.13*	2.61*	1.23*	1.03*	0.17*	0.12*	0.09*	0.07*
91		3.03	2.53	1.14	0.97	0.13	0.08	0.05	0.04

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(3) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 26 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR MANGANESE, MN IN MUNICIPAL SECONDARY EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		3.00	2.50	1.20	1.00	0.15	0.10	0.07	0.05
LAB/AN	LAB REJECTED								
11		3.00	2.50	1.21	1.01	0.14	0.09	0.06	0.08
21		3.05	2.47	1.22	1.01	0.16	0.09	< 0.06*	< 0.06*
31		2.91	2.42	1.15	0.98	0.14	0.10	0.07	0.05
41	*	3.34*	2.82*	1.31*	1.14*	0.20*	0.14*	0.08*	0.09*
51	*	2.93*	2.46*	1.11*	0.98*	0.11*	0.06*	0.02*	0.08*
62		3.11	2.65	1.36	1.07	0.19	0.13	0.06	< 0.05*
7		2.83	2.36	1.13	0.95	0.15	0.11	0.07	0.05
81		3.08	2.56	1.23	1.02	0.15	0.11	0.09	0.05
91		3.02	2.51	1.15	0.98	0.13	0.09	0.06	0.01*

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR MANGANESE, MN IN ELECTROPLATING EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		3.00	2.50	1.20	1.00	0.15	0.10	0.07	0.05
LAB/AN	LAB REJECTED								
11		3.06	2.53	1.21	1.01	0.15	0.10	0.08	0.06
21		3.06	2.58	1.25	1.03	0.16	0.12	< 0.06*	< 0.06*
31	*	2.98*	2.40*	1.15*	0.95*	0.14*	0.09*	0.06*	0.08*
41	*	3.37*	2.87*	1.29*	1.15*	0.16*	0.10*	0.08*	0.06*
51		3.20	2.56	1.19	0.99	0.15	0.10	0.07	0.05
62		3.00	2.40	1.33	1.06	0.17	0.12	0.09	< 0.05*
7		2.86	2.39*	1.15	0.98	0.16	0.12	0.08	0.06
81		3.09	2.61	1.22	1.04	0.15	0.11	0.07	0.05
91		3.03	2.63	1.18	0.96	0.14	0.10	0.07	0.06

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 27. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR MOLYBDENUM, MO IN REAGENT WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		44.80	33.60	16.80	11.20	1.68	1.12	0.84	0.56
LAB/AN	LAB REJECTED								
11	*	64.70*	55.00*	34.40*	24.80*	3.47*	2.44*	1.50*	0.96*
21		48.00	37.20	18.20	12.10	1.90	1.33	0.89	0.65
32	*	29.80*	25.60*	11.30*	8.57*	1.08*	0.89*	0.50*	0.41*
41		50.40	45.80	22.90	15.30	2.47	1.48	0.88	0.71
51		58.60	38.90	32.50	20.50	2.01	1.14	0.66	0.27
61		43.90	46.10	16.30	11.20	1.07	1.56	0.65	0.42
7		43.10	31.90	16.00	10.30	1.62	1.14	0.76	0.57
81		42.80	32.10	16.80	11.00	1.29	0.91	0.56	0.38
91	*	39.30*	31.20*	15.20*	10.60*	1.34*	0.65*	0.48*	0.35*

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR MOLYBDENUM, MO IN OHIO RIVER WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		44.80	33.60	16.80	11.20	1.68	1.12	0.84	0.56
LAB/AN	LAB REJECTED								
11	*	64.30*	45.90*	33.10*	22.30*	3.52*	2.40*	1.55*	1.06*
21		47.20	36.30	17.80	11.70	1.69	1.21	0.85	0.65
32	*	25.30*	22.10*	10.10*	7.32*	1.31*	1.01*	0.67*	0.50*
41		54.30	38.30	19.90	12.30	1.70	1.77*	0.96	0.88
51		58.10	37.60	31.90*	20.20*	1.95	1.23	0.94	0.04
61		40.90	30.90	15.70	10.50	1.59	1.04	0.68	0.46
7		41.00	29.30	16.00	10.40	1.62	1.07	0.74	0.56
81		43.80	33.30	16.70	11.30	1.56	1.15	0.67	0.50
91		40.70	30.70	15.80	10.40	1.82	1.24	0.77	0.57

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 27 (CONT.) STUDY 38, SW 846 METHOD 3010, TRACE METALS, AA FLAME

RAW DATA FOR MOLYBDENUM, MO IN MUNICIPAL SECONDARY EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		44.80	33.60	16.80	11.20	1.68	1.12	0.84	0.56
LAB/AN	LAB REJECTED								
11	*	76.80*	50.70*	35.00*	23.40*	3.55*	2.40*	1.65*	1.19*
21		47.40	35.90	17.60	11.60	1.86	1.33	0.93	0.65
32	*	32.00*	24.70*	11.60*	8.06*	1.42*	1.09*	0.71*	0.54*
41		53.20	38.80	18.70	14.70	2.40	1.73*	0.81	0.92
51		58.60	38.60	29.30*	18.80	2.08	1.18	0.97	0.18
61		44.50	34.10	15.50	10.10	1.69	1.14	0.78	0.58
7		41.70	30.90	16.40	10.90	1.73	1.18	0.86	0.59
81		43.20	32.50	16.60	11.30	1.99	1.30	0.86	0.80
91		41.60	32.70	16.20	10.30	2.04	1.33	0.89	0.59

STUDY 38, SW 846 METHOD 3010, TRACE METALS, AA FLAME

RAW DATA FOR MOLYBDENUM, MO IN ELECTROPLATING EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		44.80	33.60	16.80	11.20	1.68	1.12	0.84	0.56
LAB/AN	LAB REJECTED								
11	*	71.70*	61.00*	36.00*	24.10*	3.58*	2.40*	1.69*	1.20*
21		46.20	35.60	17.70	11.60	1.31	1.21	0.89	0.69
32		34.20	27.20	12.90	8.07*	1.68	1.19	0.85	0.59
41		51.90	35.50	16.60	11.90	1.54	1.39	1.16	1.16
51		60.10	45.30	31.90*	20.70*	2.13	1.31	0.69	0.53
61		45.60	38.80	17.90	11.50	1.62	1.01	0.62	0.36
7		40.60	29.60	16.10	10.70	1.68	1.09	0.82	0.55
81		42.50	31.80	15.90	10.80	1.54	1.10	0.69	0.74
91		43.70	33.40	16.60	10.80	1.80	1.08	0.67	0.40

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 28. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR NICKEL, NI IN REAGENT WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		5.00	4.00	2.00	1.50	0.40	0.30	0.20	0.15
LAB/AN	LAB REJECTED								
11		4.82	3.81	1.94	1.45	0.38	0.28	0.17	0.15
21		5.10	4.07	2.00	1.50	0.40	0.29	0.19	0.15
31		4.89	3.93	1.95	1.47	0.39	0.29	0.20	0.15
41		5.20	4.17	1.92	1.51	0.31	0.25	0.08	0.11
51		4.79	3.83	1.93	1.44	0.40	0.31	0.19	0.15
62		4.54	5.75*	1.94	1.90*	0.27	0.24	0.09	0.12
7		4.78	3.68	1.94	1.43	0.46	0.31	0.20	0.17
81		5.18	4.01	2.03	1.55	0.37	0.29	0.20	0.16
91	*	5.05*	4.12*	2.04*	1.56*	0.44*	0.32*	0.20*	0.19*

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR NICKEL, NI IN OHIO RIVER WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		5.00	4.00	2.00	1.50	0.40	0.30	0.20	0.15
LAB/AN	LAB REJECTED								
11		4.74	3.75	1.36	1.40	0.34	0.28	0.17	0.13
21		4.99	3.95	2.01	1.49	0.40	0.29	0.21	0.16
31		4.83	3.87	1.98	1.46	0.38	0.29	0.20	0.15
41		4.96	4.08	1.96	1.31	0.38	0.20*	0.19	0.18
51		4.76	3.74	1.87	1.43	0.37	0.28	0.18	0.12
62		4.41	3.36	1.93	1.51	0.39	0.09*	0.15	0.06
7		4.75	3.97	1.97	1.49	0.42	0.30	0.21	0.12
81		5.24	4.12	2.01	1.51	0.36	0.28	0.18	0.12
91		5.12	4.08	2.03	1.55	0.39	0.28	0.21	0.15

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS:
1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 28 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR NICKEL, NI IN MUNICIPAL SECONDARY EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		5.00	4.00	2.00	1.50	0.40	0.30	0.20	0.15
LAB/AN	LAB REJECTED								
11		4.69	3.76	1.92	1.43	0.39	0.29	0.20	0.17
21		5.17	3.95	1.97	1.47	0.40	0.24	0.18	0.10
31		4.84	3.92	1.95	1.49	0.40	0.30	0.20	0.15
41		4.98	3.79	2.14	1.74	0.46	0.22	0.20	0.20
51	*	4.49*	3.69*	1.80*	1.41*	0.32*	0.26*	0.16*	0.09*
62	*	5.50*	4.45*	3.42*	2.72*	1.69*	1.66*	1.72*	1.81*
7		5.11	4.55	2.13	1.40	0.41	0.30	0.19	0.08
81		5.01	4.19	2.07	1.56	0.46	0.38	0.26	0.24
91		5.18	4.05	1.98	1.53	0.36	0.16	0.10	0.09

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR NICKEL, NI IN ELECTROPLATING EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		5.30	4.00	2.00	1.50	0.40	0.30	0.20	0.15
LAB/AN	LAB REJECTED								
11		4.35	3.84	1.93	1.45	0.37	0.30	0.18	0.14
21		5.00	4.02	1.94	1.46	0.36	0.27	0.16	0.14
31		4.86	3.86	1.97	1.51	0.40	0.29	0.18	0.13
41		5.46	4.43	2.11	1.48	0.47	0.25	0.25	0.22
51	*	4.63*	3.76*	1.85*	1.36*	0.35*	0.27*	0.19*	0.12*
62	*	5.23*	4.62*	2.35*	1.54*	0.54*	0.39*	0.30*	0.30*
7		4.76	4.06	1.98	1.57	0.45	0.29	0.26	0.21
81		5.18	4.11	2.03	1.56	0.39	0.29	0.18	0.13
91		4.99	4.08	2.08	1.51	0.36	0.34	0.21	0.17

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 29. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR POTASSIUM, K IN REAGENT WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		30.00	24.00	15.00	12.00	6.00	4.80	3.00	2.40
LAB/AN	LAB REJECTED								
11		27.70	20.60	13.10	10.20	5.60	4.00	2.50	1.90
21	*	31.10*	25.30*	18.00*	14.40*	6.76*	6.40*	3.73*	3.18*
31		28.70	21.50	11.90	9.42	6.35	4.48	2.37	2.05
41		26.50	20.30	13.10	9.98	4.95	4.95	2.54	1.89
51		28.60	23.00	14.50	11.70	5.52	4.35	2.52	2.37
62	*	19.70*	19.30*	7.93*	6.58*	0.69*	0.90*	0.41*	0.22*
7		28.30	22.10	14.00	11.30	6.06	5.02	2.81	2.16
81		30.90	24.40	16.80	12.20	5.93	4.63	3.01	2.43
91		30.70	24.10	14.90	9.89	5.90	4.85	3.07	2.40

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR POTASSIUM, K IN OHIO RIVER WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		30.00	24.00	15.00	12.00	6.00	4.80	3.00	2.40
LAB/AN	LAB REJECTED								
11		26.30	22.20	13.50	10.70	5.70	4.60	2.80	2.30
21	*	30.10*	23.40*	17.30*	13.90*	6.70*	5.44*	3.35*	2.75*
31		25.10	20.30	14.10	10.70	5.14	4.47	2.70	1.97
41	*	27.60*	20.60*	13.00*	4.54*	4.90*	4.05*	2.54*	1.76*
51		28.90	23.30	14.40	11.50	5.61	4.74	2.83	2.07
62	*	15.90*	15.30*	7.85*	6.23*	6.24*	0.32*	0.04*	0.08*
7		27.60	22.40	14.60	10.80	5.54	2.57*	2.92	2.39
81		31.00	23.10	16.10	12.70	6.08	4.60	2.86	2.52
91		30.40	23.50	15.20	11.20	5.82	4.07	2.18*	1.83

* = REJECTED

CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 29 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR POTASSIUM, K IN MUNICIPAL SECONDARY EFFLUENT

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		30.00	24.00	15.00	12.00	6.00	4.80	3.00	2.40
11		25.80	20.10	13.60	10.70	5.50	4.20	3.00	2.30
21		28.80	23.10	13.90	11.10	6.23	6.28	4.00*	3.07
31	*	26.50*	20.10*	12.70*	10.10*	4.65*	3.95*	2.12*	1.61*
41		34.30	25.40	15.10	10.60	5.98	5.58	2.78	1.78
51		27.90	22.60	14.80	11.80	5.69	4.85	2.84	2.46
62		18.40	17.30	12.70	12.20	3.93*	3.89	5.97*	4.97*
7		30.60	24.60	14.30	11.50	6.11	4.38	2.72	2.01
81		29.40	24.20	15.60	12.00	5.77	4.37	2.47	2.77
91	*	30.30*	24.80*	16.80*	14.00*	7.46*	5.99*	4.59*	3.17*

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR POTASSIUM, K IN ELECTROPLATING EFFLUENT

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		30.00	24.00	15.00	12.00	6.00	4.80	3.00	2.40
11		26.00	23.30	16.20	10.30	6.30	4.90	3.80	3.50
21		28.30	22.60	14.00	10.60	6.31	3.84	3.78	2.51
31	*	26.70*	21.30*	12.90*	9.80*	5.29*	4.90*	2.06*	1.56*
41		27.70	22.10	14.90	12.10	4.70	3.10	2.90	3.70
51		27.50	21.70	14.20	11.50	5.41	4.94	2.86	2.26
62		15.00*	13.10*	9.19*	7.45	4.92	3.34	3.59	3.84
7		29.40	23.80	15.30	12.50	6.06	5.25	3.08	1.99
81		32.80	24.70	16.90	12.60	5.80	4.80	3.00	2.30
91	*	32.20*	27.30*	18.10*	15.70*	9.56*	7.03*	6.91*	5.23*

* = REJECTED

CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 30. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR SILVER, AG IN REAGENT WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		4.00	3.00	1.50	1.00	0.15	0.10	0.07	0.05
LAB/AN	LAB REJECTED								
11		0.98	0.83	0.80	0.69	0.13	0.10	0.08	0.07
21		1.31	0.72	1.25	1.01	0.16	0.10	0.07	0.06
31		1.33	1.23	1.34	0.87	0.13	0.08	0.06	0.04
41		0.68	0.76	0.37	0.60	0.14	0.10	0.08	0.05
51		0.95	1.06	0.15	0.19	0.18	0.12	0.11	0.10
62		0.88	1.18	1.32	0.95	0.15	0.11	0.08	0.08
7		0.45	0.42	0.33	0.47	0.15	0.10	0.07	0.05
81		0.20	0.19	0.20	0.40	0.14	0.09	0.07	0.05
91		0.55	1.86	1.55	1.03	0.11	0.08	0.06	0.04

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR SILVER, AG IN OHIO RIVER WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		4.00	3.00	1.50	1.00	0.15	0.10	0.07	0.05
LAB/AN	LAB REJECTED								
11		0.32	0.80	0.74	0.56	0.09	0.08	0.06	0.04
21	*	2.00*	1.82*	1.38*	0.90*	0.15*	0.10*	0.08*	0.06*
31		0.85	0.99	0.51	0.50	0.16	0.10	0.07	0.05
41		0.45	0.30	0.19	0.15	0.08	0.08	0.07	0.05
51		0.14	0.15	0.13	0.12	0.09	0.08	0.09	0.10
62	*	0.22*	0.19*	0.16*	0.12*	0.04*	0.04*	0.04*	0.04*
7		0.36	0.57	0.37	0.55	0.16	0.11	0.09	0.09
81		0.41	0.27	0.17	0.11	0.04	0.05	0.05	0.05
91		0.56	0.38	0.23	0.19	0.07	0.07	0.06	0.05

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 30 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR SILVER, AG IN MUNICIPAL SECONDARY EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		4.00	3.00	1.50	1.00	0.15	0.10	0.07	0.05
LAB/AN	LAB REJECTED								
11		1.33	1.02	0.91	0.77	0.13	0.12	0.09	0.07
21		1.95	2.15	1.24	0.77	0.12	0.08	0.05	0.05
31		0.87	1.27	0.68	0.71	0.13	0.08	0.06	0.04
41		0.90	0.57	0.41	0.45	0.12	0.08	0.06	0.07
51		0.21	0.28	0.16	0.12	0.11	0.10	0.27*	0.06
62		1.04	1.46	0.99	0.58	0.12	0.08	0.04	0.04
7		0.13	0.13	0.11	0.34	0.17*	0.12	0.10	0.10
81		0.88	0.95	0.28	0.55	0.11	0.07	0.05	0.04
91		0.72	1.77	0.30	0.70	0.13	0.09	0.04	0.04

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR SILVER, AG IN ELECTROPLATING EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		4.00	3.00	1.50	1.00	0.15	0.10	0.07	0.05
LAB/AN	LAB REJECTED								
11		1.14*	0.97*	0.84*	0.78	0.12	0.10	0.05	0.02
21	*	1.91*	1.60*	1.36*	1.05*	0.14*	0.10*	0.07*	0.05*
31	*	1.23*	1.79*	1.26*	1.09*	0.13*	0.10*	0.06*	0.05*
41		0.08	0.08	0.07	0.07	0.05	0.06	0.04	0.07
51		0.05	0.05	0.04	0.45	0.02	0.03	0.04	0.04
62	*	0.05*	0.03*	< 0.02*	0.03*	< 0.02*	< 0.02*	< 0.02*	< 0.02*
7		0.26*	0.36*	0.28*	0.38	0.15	0.14	0.09*	0.13
81		0.10	0.07	0.03	0.04	0.02	0.03	0.02	0.03
91		0.08	0.09	0.08	0.08	0.04	0.04	0.03	0.04

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

Table 31. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR SODIUM, NA IN REAGENT WATER

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		600.00	480.00	300.00	240.00	120.00	96.00	36.00	24.00
11		532.00	437.00	269.00	229.00	124.00	102.00	35.60	23.70
21		615.00	491.00	316.00	252.00	134.00	107.00	40.80	26.90
31		633.00	503.00	303.00	243.00	121.00	95.00	36.00	23.00
41		640.00	515.00	336.00	247.00	123.00	104.00	38.80	27.10
51		591.00	482.00	295.00	234.00	117.00	93.80	34.80	23.90
62		706.00	794.00*	331.00	285.00	108.00	137.00*	40.00	26.30
7		606.00	466.00	314.00	252.00	112.00	92.10	38.20	25.50
81		628.00	452.00	303.00	239.00	116.00	94.30	37.50	26.80
91		627.00	516.00	317.00	261.00	132.00	105.00	38.90	26.70

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR SODIUM, NA IN OHIO RIVER WATER

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		600.00	480.00	300.00	240.00	120.00	96.00	36.00	24.00
11		550.00	446.00	289.00	244.00	123.00	105.00	38.10	26.10
21		591.00	875.00*	300.00	242.00	120.00	96.80	37.30	25.80
31	*	601.00*	443.00*	278.00*	224.00*	109.00*	88.00*	33.00*	23.00*
41		583.00	514.00	344.00	260.00	120.00	101.00	38.40	26.50
51		599.00	477.00	287.00	236.00	119.00	95.50	35.60	23.80
62		702.00*	512.00	354.00	262.00	137.00	102.00	34.80	22.20
7		568.00	448.00	303.00	250.00	110.00	86.70	32.90	22.10
81		612.00	488.00	305.00	249.00	120.00	94.50	36.10	25.40
91	*	647.00*	528.00*	323.00*	269.00*	133.00*	108.00*	38.00*	25.80*

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 31 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR SODIUM, NA IN MUNICIPAL SECONDARY EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		600.00	480.00	300.00	240.00	120.00	96.00	36.00	24.00
LAB/AN	LAB REJECTED								
11		660.00	388.00	182.00*	163.00*	110.00	98.50	38.60	28.70
21		598.00	468.00	293.00	237.00	114.00	92.60	31.40	20.20
31	*	563.00*	451.00*	270.00*	222.00*	114.00*	88.00*	31.00*	20.00*
41	*	641.00*	524.00*	331.00*	274.00*	145.00*	107.00*	55.00*	40.00*
51		563.00	473.00	299.00	238.00	117.00	96.00	33.30	22.50
62	*	671.00*	530.00*	325.00*	251.00*	176.00*	106.00*	105.00*	26.70*
7	*	579.00*	454.00*	283.00*	227.00*	97.80*	80.60*	27.20*	17.60*
81		591.00	502.00	320.00	242.00	123.00	102.00	38.90	27.80
91		641.00	517.00	322.00	260.00	135.00	100.00	38.00	21.00

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR SODIUM, NA IN ELECTROPLATING EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		600.00	480.00	300.00	240.00	120.00	96.00	36.00	24.00
LAB/AN	LAB REJECTED								
11		597.00	477.00	96.90*	81.90*	51.70*	49.40*	40.90	28.90
21	*	862.00*	747.00*	561.00*	499.00*	383.00*	358.00*	294.00*	286.00*
31	*	537.00*	420.00*	255.00*	201.00*	101.00*	77.00*	24.00*	18.00*
41		601.00	514.00	272.00	228.00	120.00	88.00	26.00	24.00
51		587.00	448.00	288.00	233.00	109.00	94.50	39.30	23.00
62	*	690.00*	557.00*	457.00*	434.00*	348.00*	303.00*	263.00*	264.00*
7	*	537.00*	440.00*	246.00*	202.00*	87.60*	66.10*	21.30*	19.80*
81		640.00*	476.00	265.00	219.00	116.00	96.50	36.20	36.40
91		592.00	521.00	320.00	225.00	121.00	104.00	31.00	20.00

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 32. STUDY 39,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR THALLIUM, TI IN REAGENT WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		20.00	16.00	10.00	8.00	1.20	1.00	0.60	0.48
LAB/AN	LAB REJECTED								
11		20.70	16.70	10.70	8.53	1.35	0.80	0.45	0.28
21		20.60	16.50	10.40	8.39	1.18	0.92	0.49	0.36
31		19.50	15.80	9.97	7.96	1.15	0.95	0.56	0.43
41		20.90	17.20	10.80	8.32	1.27	1.09	0.82	0.60
51		20.40	16.40	10.10	8.01	1.18	0.96	0.58	0.44
62		20.40	23.10*	9.66	8.08	0.72*	1.03	0.30	0.08
7		20.20	16.00	10.00	8.18	1.32	1.05	0.61	0.44
81		21.30	16.60	10.60	8.37	1.15	1.03	< 0.81*	< 0.81*
91		20.90	17.20	10.70	8.49	1.29	1.00	0.54	0.36

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR THALLIUM, TI IN OHIO RIVER WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		20.00	16.00	10.00	8.00	1.20	1.00	0.60	0.48
LAB/AN	LAB REJECTED								
11		20.40	16.40	10.40	8.42	1.26	1.09	0.63	0.50
21	*	19.30*	15.70*	10.00*	7.87*	1.09*	0.88*	0.49*	0.36*
31		20.00	16.30	10.50	7.97	1.14	0.96	0.57	0.41
41	*	21.30*	16.90*	11.00*	8.23*	1.27*	0.94*	0.66*	0.57*
51		20.50	16.30	10.20	8.11	1.26	1.11	0.69	0.51
62	*	18.60*	15.10*	10.50*	7.66*	1.03*	0.93*	0.30*	< 0.10*
7		20.20	16.00	9.91	8.07	1.25	0.98	0.62	0.54
81		21.40	16.60	10.20	8.10	1.00	0.99	< 0.72*	< 0.72*
91		21.40	16.30	10.60	8.53	1.21	1.00	0.58	0.42

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 32 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR THALLIUM, TI IN MUNICIPAL SECONDARY EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		20.00	16.00	10.00	8.00	1.20	1.00	0.60	0.48
LAB/AN	LAB REJECTED								
11	*	20.60*	16.50*	10.50*	8.36*	1.32*	1.10*	0.71*	0.56*
21		20.40	15.90	10.00	7.91	1.14	0.92	0.49	0.36
31		20.50	16.10	10.10	8.20	1.21	0.98	0.56	0.45
41		21.10	16.80	9.98	7.98	1.21	0.91	0.54	0.27
51		20.20	16.60	10.30	8.27	1.28	1.06	0.67	0.56
62		19.90	16.10	10.10	8.40	1.14	0.61*	0.72	< 0.10*
7		20.00	16.10	10.20	8.18	1.29	1.03	0.60	0.52
81		20.70	16.30	10.70	8.21	1.21	1.04	< 0.74*	< 0.74*
91		21.40	16.70	10.60	8.13	1.13	0.88	0.46	0.28

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR THALLIUM, TI IN ELECTROPLATING EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		20.00	16.00	10.00	8.00	1.20	1.00	0.60	0.48
LAB/AN	LAB REJECTED								
11		20.70	16.70	10.40	8.47	1.30	1.10	0.70	0.55
21		20.40	16.30	10.10	8.04	1.09	0.92	0.44	0.40*
31		20.00	15.90	9.90	8.22	1.19	0.99	0.60	0.50
41		20.80	16.60	10.60	8.17	1.30	0.91	0.79	0.54
51		20.30	16.20	10.20	8.24	1.28	1.11	0.68	0.54
62		19.70	16.70	10.30	8.11	1.26	0.95	0.63	0.21*
7		19.70	15.90	10.00	8.26	1.23	0.98	0.57	0.49
81		21.50	17.50	10.70	8.55	1.22	1.10	< 0.72*	< 0.72*
91		21.10	17.70	10.50	8.24	1.28	1.13	0.65	0.54

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 33. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR VANADIUM, V IN REAGENT WATER

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		99.00	81.00	45.00	36.00	2.70	2.25	1.35	1.08
11		101.00	80.40	44.50	35.00	2.64	2.18	1.32	1.02
21		106.00	86.70	48.40	38.70	3.10	2.63	1.51	1.14
31	*	133.00*	109.00*	59.50*	47.30*	3.10*	2.60*	1.60*	1.30*
41	*	79.80*	73.70*	41.10*	35.00*	2.45*	2.94*	< 0.64*	< 0.64*
51		128.00*	104.00	53.40	46.10*	4.59*	2.36	1.24	0.95
61		102.00	118.00	46.20	38.20	2.31	2.62	1.31	1.11
7		94.60	79.70	45.00	34.30	2.97	2.43	1.62	1.35
81		109.00	87.00	48.50	38.40	2.90	2.30	1.50	1.20
91		107.00	83.50	44.10	37.60	2.85	2.30	1.35	1.04

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR VANADIUM, V IN OHIO RIVER WATER

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		99.00	81.00	45.00	36.00	2.70	2.25	1.35	1.08
11		97.00	81.40	43.90	35.30	2.69	2.25	1.35	1.09
21		100.00	82.00	46.20	36.80	2.82	2.45	1.42	1.14
31	*	131.00*	109.00*	58.20*	47.00*	3.30*	2.70*	1.50*	1.20*
41		126.00	103.00	54.60	39.70	3.26	1.81*	1.29	0.56*
51		146.00	117.00	54.60	50.50*	3.79	2.36	1.22	0.98
61	*	94.30*	78.00*	44.00*	33.40*	2.40*	1.90*	1.00*	0.70*
7		94.40	76.40	44.10	34.40	2.78	2.36	1.38	1.11
81		111.00	90.00	49.50	39.90	3.00	2.50	1.50	1.30
91		106.00	80.40	42.90	39.70	3.04	2.46	1.54	1.12

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 33 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR VANADIUM, V IN MUNICIPAL SECONDARY EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		99.00	81.00	45.00	36.00	2.70	2.25	1.35	1.08
LAB/AN	LAB REJECTED								
11		93.50	76.10	43.30	38.80	2.61	2.20	1.29	1.09
21		103.00	82.30	46.00	36.20	2.72	2.26	1.33	1.05
31	*	136.00*	108.00*	60.50*	45.30*	3.30*	2.40*	1.40*	1.20*
41	*	86.20*	75.00*	41.00*	32.00*	1.79*	2.37*	0.96*	0.78*
51		131.00	107.00	54.00	48.90	4.30*	2.20	1.09	0.73
61		92.20	176.00*	43.20	33.50	2.62	2.01	1.11	0.81
7		91.00	74.00	44.20	38.80	2.73	2.38	1.43	1.17
81		108.00	93.00	50.10	40.00	3.10	2.50	1.50	1.20
.91		105.00	82.70	48.90	40.10	3.05	2.43	1.53	0.89

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR VANADIUM, V IN ELECTROPLATING EFFLUENT

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		99.00	81.00	45.00	36.00	2.70	2.25	1.35	1.08
LAB/AN	LAB REJECTED								
11		95.50	79.00	43.80	38.90	2.59	2.17	1.30	1.08
21		101.00	86.30	46.10	37.50	2.82	2.35	1.33	1.05
31	*	135.00*	111.00*	60.80*	48.00*	3.30*	2.70*	1.50*	1.30*
41	*	82.50*	64.50*	36.10*	26.40*	2.01*	1.39*	0.92*	0.71*
51		134.00*	106.00	56.60	49.10*	2.89	2.32	1.17	0.68
61	*	93.00*	77.00*	43.00*	32.60*	2.00*	1.90*	1.00*	0.70*
7		98.50	80.90	44.60	35.30	3.09	2.65	1.62	1.32
81		105.00	88.50	48.20	39.50	2.90	2.40	1.40	1.30
91		106.00	90.00	50.10	39.40	2.89	2.55	1.53	1.17

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 34. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR ZINC, ZN IN REAGENT WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		1.00	0.80	0.50	0.40	0.15	0.10	0.06	0.05
LAB/AN	LAB REJECTED								
11	*	0.93*	0.72*	0.46*	0.35*	0.13*	0.09*	0.05*	0.08*
21		1.01	0.80	0.50	0.40	0.16	0.11	0.06	0.05
31	*	0.94*	0.73*	0.47*	0.36*	0.14*	0.07*	0.05*	0.04*
41	*	1.07*	0.88*	0.52*	0.45*	0.20*	0.12*	0.08*	0.08*
51		1.05	0.85	0.51	0.42	0.16	0.11	0.07	0.05
62	*	1.01*	1.24*	0.59*	0.51*	0.17*	0.12*	0.06*	0.05*
7		0.95	0.78	0.46	0.37	0.15	0.10	0.06	0.04
81		0.99	0.76	0.48	0.38	0.14	0.09	0.05	0.04
91		1.00	0.78	0.49	0.40	0.19	0.10	0.05	0.04

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR ZINC, ZN IN OHIO RIVER WATER

		AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		1.00	0.80	0.50	0.40	0.15	0.10	0.06	0.05
LAB/AN	LAB REJECTED								
11		0.89	0.68	0.40	0.33	0.08	0.08	0.08*	0.02*
21	*	0.30*	0.59*	0.32*	0.21*	< 0.00*	< 0.00*	< 0.00*	< 0.00*
31		0.87	0.69	0.40	0.30	0.10	0.06	0.03*	0.03*
41		0.91	0.82	0.32	0.37	0.10	0.02	< 0.02*	< 0.02*
51	*	0.59*	0.51*	0.15*	0.05*	< 0.00*	< 0.00*	< 0.00*	< 0.00*
62	*	2.36*	2.19*	1.71*	1.70*	1.46*	1.43*	1.41*	1.42*
7		0.94	0.77	0.55	0.45	0.07	0.01	< 0.01*	< 0.01*
81	*	1.59*	1.37*	1.06*	0.90*	0.71*	0.68*	0.06*	0.65*
91		0.34	0.65	0.42	0.33	0.09	0.01	- 0.01*	- 0.02*

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

TABLE 34 (CONT.) STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR ZINC, ZN IN MUNICIPAL SECONDARY EFFLUENT

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		1.00	0.80	0.50	0.40	0.15	0.10	0.06	0.05
11		0.87	0.70	0.45	0.37	0.13	0.10	0.04	0.04
21		1.06	0.79	0.50	0.39	0.15	0.12	0.04	0.03
31		0.96	0.79	0.47	0.38	0.14	0.09	0.05	0.03
41		1.02	0.97	0.43	0.37	0.24	0.05	< 0.02*	< 0.02*
51	*	0.59*	0.49*	0.16*	0.05*	< 0.00*	< 0.00*	< 0.00*	< 0.00*
62	*	3.36*	2.34*	1.94*	1.72*	1.60*	1.55*	1.54*	1.50*
7		0.98	0.80	0.51	0.44	0.18	0.11	0.07	0.07
81	*	1.12*	1.04*	0.84*	0.67*	0.36*	0.42*	0.41*	0.41*
91		1.10	0.74	0.53	0.46	0.23	0.23	0.14	- 0.17*

STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

RAW DATA FOR ZINC, ZN IN ELECTROPLATING EFFLUENT

LAB/AN	LAB REJECTED	AMPUL 1	AMPUL 2	AMPUL 3	AMPUL 4	AMPUL 5	AMPUL 6	AMPUL 7	AMPUL 8
INCREMENT IN UG/ML		1.00	0.80	0.50	0.40	0.15	0.10	0.06	0.05
11		0.95	0.75	0.45	0.37	0.10	0.09	0.04	0.04
21		0.99	0.80	0.52	0.42	0.17	0.11	0.06	0.06
31		0.97	0.76	0.47	0.36	0.13	0.05	0.02	0.02
41		0.89	0.72	0.51	0.40	0.17	0.13	0.03	< 0.02*
51		0.97	0.71	0.57	0.43	0.14	0.08	0.06	0.05
62	*	2.54*	1.22*	0.80*	0.61*	0.42*	0.43*	0.38*	0.40*
7		0.91	0.76	0.45	0.38	0.15	0.07	< 0.01*	< 0.01*
81		0.84	0.71	0.46	0.45	0.20	0.11	0.07	0.09
91		0.89	0.75	0.50	0.37	0.15	0.12	0.07	0.09

* = REJECTED
CT = CONCENTRATION, DM = DIFFERENT METHOD, IM = IMPROPER METHOD, UD = UNACCEPTABLE DATA, < = LESS THAN

CURRENT SIGNIFICANCE LEVELS: 1. LAB RANKING DATA REJECTION TESTS, ALPHA =0.05
2. INDIVIDUAL OUTLIER TESTS USING THOMPSON(S) RULE, ALPHA =0.05

AMPUL = SAMPLE

APPENDIX C

STATISTICAL SUMMARIES

TABLE 35. STUDY 38, SW 846 METHOD 3010, TRACE METALS, AA FLAME

STATISTICAL SUMMARY FOR ALUMINUM, AL ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	7	7	7	7	6	7	6	6
TRUE CONC (C) UG/ML	50.00	40.00	50.00	40.00	50.00	40.00	50.00	40.00
MEAN RECOVERY (X)	51.67	40.74	48.07	37.36	49.43	37.61	49.02	40.02
% RECOVERY	103.34	101.86	96.14	93.39	98.87	94.08	98.03	100.04
OVERALL STD DEV (S)	3.28	6.05	3.12	2.87	1.13	3.69	2.95	1.67
OVERALL REL STD DEV, %	6.34	14.85	6.49	7.67	2.29	9.81	6.01	4.19
SINGLE STD DEV, (SR)	4.20		1.45		2.54		1.10	
ANALYST REL DEV, %	9.09		3.40		5.83		2.48	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	7	7	7	7	7	7	5	7
TRUE CONC (C) UG/ML	25.00	20.00	25.00	20.00	25.00	20.00	25.00	20.00
MEAN RECOVERY (X)	24.74	19.90	23.73	18.96	22.99	18.76	25.22	21.04
% RECOVERY	98.97	99.50	94.91	94.79	91.94	93.79	100.88	105.21
OVERALL STD DEV (S)	1.93	1.25	1.33	1.10	2.74	2.30	0.63	2.93
OVERALL REL STD DEV, %	7.81	6.26	5.62	5.81	11.92	12.27	2.48	13.90
SINGLE STD DEV, (SR)	0.68		0.21		0.72		0.92	
ANALYST REL DEV, %	3.05		0.99		3.47		3.98	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	7	7	7	7	7	7	7	6
TRUE CONC (C) UG/ML	6.00	5.00	6.00	5.00	6.00	5.00	6.00	5.00
MEAN RECOVERY (X)	5.64	4.88	5.35	4.56	5.51	4.59	6.08	4.80
% RECOVERY	93.95	97.60	89.10	91.23	91.30	91.71	101.26	95.93
OVERALL STD DEV (S)	0.43	0.40	0.29	0.30	0.57	0.46	0.93	0.25
OVERALL REL STD DEV, %	7.65	8.26	5.39	6.60	10.32	10.13	15.32	5.22
SINGLE STD DEV, (SR)	0.50		0.20		0.18		0.19	
ANALYST REL DEV, %	9.54		4.02		3.55		3.42	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	7	7	7	7	7	7	7	7
TRUE CONC (C) UG/ML	3.00	2.50	3.00	2.50	3.00	2.50	3.00	2.50
MEAN RECOVERY (X)	2.85	2.26	2.56	2.08	2.74	2.39	2.86	2.16
% RECOVERY	95.14	90.34	85.24	83.14	91.19	95.77	95.33	86.57
OVERALL STD DEV (S)	0.23	0.22	0.22	0.33	0.33	0.34	0.50	0.51
OVERALL REL STD DEV, %	7.92	9.89	8.67	15.81	12.01	14.39	17.52	23.69
SINGLE STD DEV, (SR)	0.18		0.15		0.17		0.22	
ANALYST REL DEV, %	7.20		6.30		6.71		8.83	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 36. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

STATISTICAL SUMMARY FOR ANTIMONY, SB ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	9	8	8	8	7	7	6	6
TRUE CONC (C) UG/ML	40.00	32.00	40.00	32.00	40.00	32.00	40.00	32.00
MEAN RECOVERY (X)	43.16	33.89	43.27	35.24	41.87	34.46	41.98	33.72
% RECOVERY	107.89	105.90	108.19	110.12	104.68	107.68	104.96	105.36
OVERALL STD DEV (S)	3.30	2.97	4.65	3.28	1.48	2.32	0.57	0.37
OVERALL REL STD DEV, %	7.66	8.76	10.74	9.31	3.54	6.72	1.37	1.10
SINGLE STD DEV, (SR)	1.40		2.00		1.33		0.25	
ANALYST REL DEV, %	3.64		5.09		3.50		0.65	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	8	8	7	8	7	6	6	6
TRUE CONC (C) UG/ML	16.00	12.00	16.00	12.00	16.00	12.00	16.00	12.00
MEAN RECOVERY (X)	16.36	12.46	16.16	11.67	16.49	12.07	16.72	12.57
% RECOVERY	102.27	103.85	100.98	97.29	103.08	100.56	104.48	104.72
OVERALL STD DEV (S)	0.52	0.18	0.56	0.63	1.05	0.44	0.18	0.25
OVERALL REL STD DEV, %	3.17	1.48	3.48	5.41	6.35	3.62	1.10	1.99
SINGLE STD DEV, (SR)	0.37		0.31		0.21		0.10	
ANALYST REL DEV, %	2.60		2.20		1.45		0.67	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	8	9	8	8	7	7	7	7
TRUE CONC (C) UG/ML	1.60	1.20	1.60	1.20	1.60	1.20	1.60	1.20
MEAN RECOVERY (X)	1.29	1.10	1.41	1.03	1.53	1.09	1.75	1.25
% RECOVERY	80.78	91.57	88.05	86.04	95.36	90.83	109.11	103.93
OVERALL STD DEV (S)	0.25	0.26	0.23	0.16	0.16	0.20	0.23	0.19
OVERALL REL STD DEV, %	19.09	23.43	16.16	15.84	10.63	18.52	13.17	15.07
SINGLE STD DEV, (SR)	0.22		0.15		0.17		0.07	
ANALYST REL DEV, %	18.26		12.21		13.33		4.91	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	7	7	6	5	6	3	7	6
TRUE CONC (C) UG/ML	0.80	0.56	0.80	0.56	0.80	0.56	0.80	0.56
MEAN RECOVERY (X)	0.69	0.39	0.78	0.48	0.77	0.39	0.87	0.54
% RECOVERY	86.61	70.41	97.71	85.00	95.83	70.24	108.57	96.13
OVERALL STD DEV (S)	0.07	0.18	0.11	0.09	0.18	0.25	0.16	0.07
OVERALL REL STD DEV, %	10.09	46.83	13.52	17.99	23.73	63.98	18.44	12.73
SINGLE STD DEV, (SR)	0.09		0.03		0.08		0.08	
ANALYST REL DEV, %	17.22		4.90		13.85		11.15	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 37. STUDY 38, SW 846 METHOD 3010, TRACE METALS, AA FLAME

STATISTICAL SUMMARY FOR BARIUM, BA ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	8	8	7	6	8	8	6	7
TRUE CONC (C) UG/ML	20.00	16.00	20.00	16.00	20.00	16.00	20.00	16.00
MEAN RECOVERY (X)	17.89	15.19	16.84	16.32	7.96	7.73	1.86	3.40
% RECOVERY	89.44	94.92	84.21	101.98	39.79	48.30	9.31	21.28
OVERALL STD DEV (S)	3.31	1.92	5.04	1.04	4.46	3.32	1.14	3.87
OVERALL REL STD DEV, %	18.48	12.63	29.92	6.36	56.11	42.96	61.21	113.62
SINGLE STD DEV, (SR)	1.39		2.14		1.67		0.68	
ANALYST REL DEV, %	8.41		12.94		21.27		25.82	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	8	8	7	7	7	8	7	7
TRUE CONC (C) UG/ML	8.00	6.00	8.00	6.00	8.00	6.00	8.00	6.00
MEAN RECOVERY (X)	7.75	5.93	8.33	6.13	6.55	5.36	3.24	4.42
% RECOVERY	96.92	98.87	104.07	102.12	81.87	89.40	40.52	73.64
OVERALL STD DEV (S)	0.95	0.76	0.60	0.42	0.78	0.59	2.38	2.01
OVERALL REL STD DEV, %	12.27	12.87	7.24	6.82	11.88	10.98	73.38	45.46
SINGLE STD DEV, (SR)	0.34		0.28		0.49		0.98	
ANALYST REL DEV, %	4.95		3.84		8.28		25.52	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	8	8	6	6	8	7	7	7
TRUE CONC (C) UG/ML	1.20	1.00	1.20	1.00	1.20	1.00	1.20	1.00
MEAN RECOVERY (X)	1.20	0.97	1.23	1.04	1.37	1.17	1.26	1.06
% RECOVERY	100.31	97.50	102.22	104.33	113.85	117.43	105.12	106.43
OVERALL STD DEV (S)	0.12	0.13	0.06	0.09	0.19	0.08	0.17	0.17
OVERALL REL STD DEV, %	9.56	13.11	5.27	8.33	14.12	6.58	13.67	15.77
SINGLE STD DEV, (SR)	0.06		0.05		0.08		0.03	
ANALYST REL DEV, %	5.39		4.54		6.33		2.62	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	7	8	7	7	8	8	7	7
TRUE CONC (C) UG/ML	0.60	0.52	0.60	0.52	0.60	0.52	0.60	0.52
MEAN RECOVERY (X)	0.57	0.51	0.55	0.46	0.70	0.60	0.58	0.54
% RECOVERY	95.71	98.80	91.67	88.19	116.67	115.87	96.67	103.57
OVERALL STD DEV (S)	0.06	0.06	0.17	0.20	0.08	0.16	0.13	0.11
OVERALL REL STD DEV, %	10.54	11.77	30.98	43.59	11.45	25.74	21.97	20.99
SINGLE STD DEV, (SR)	0.05		0.04		0.08		0.07	
ANALYST REL DEV, %	9.03		7.39		12.09		12.00	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 38. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

STATISTICAL SUMMARY FOR BERYLLIUM, BE ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	7	6	6	6	6	6	4	5
TRUE CONC (C) UG/ML	2.00	1.60	2.00	1.60	2.00	1.60	2.00-	1.60
MEAN RECOVERY (X)	1.75	1.41	1.92	1.53	1.94	1.54	1.98	1.54
% RECOVERY	87.71	88.23	96.00	95.62	97.25	96.46	99.25	96.25
OVERALL STD DEV (S)	0.23	0.21	0.12	0.06	0.11	0.07	0.03	0.07
OVERALL REL STD DEV, %	13.29	14.71	5.99	4.19	5.41	4.74	1.33	4.57
SINGLE STD DEV, (SR)	0.04		0.05		0.04		0.04	
ANALYST REL DEV, %	2.42		2.69		2.04		2.06	
HIGH-MEDIUM YOUDEN P	3	4	3	8	3	4	3	4
NUMBER OF DATA POINTS	7	7	6	7	6	6	5	5
TRUE CONC (C) UG/ML	0.80	0.60	0.80	0.60	0.80	0.60	0.80	0.60
MEAN RECOVERY (X)	0.71	0.53	0.76	0.55	0.78	0.58	0.75	0.58
% RECOVERY	88.57	88.81	94.58	91.90	98.12	96.11	94.00	96.33
OVERALL STD DEV (S)	0.08	0.05	0.04	0.04	0.05	0.03	0.04	0.02
OVERALL REL STD DEV, %	10.88	9.05	4.92	8.07	5.91	5.10	4.83	3.95
SINGLE STD DEV, (SR)	0.02		0.01		0.03		0.01	
ANALYST REL DEV, %	3.47		1.44		3.91		1.78	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	7	5	7	6	5	5	5	3
TRUE CONC (C) UG/ML	0.06	0.04	0.06	0.04	0.06	0.04	0.06	0.04
MEAN RECOVERY (X)	0.05	0.04	0.05	0.04	0.06	0.04	0.06	0.04
% RECOVERY	81.19	104.00	86.19	91.25	97.67	97.50	94.33	97.50
OVERALL STD DEV (S)	0.02	0.00	0.01	0.00	0.00	0.00	0.01	0.00
OVERALL REL STD DEV, %	31.97	4.02	23.68	9.76	3.33	10.09	9.56	UND
SINGLE STD DEV, (SR)	0.01		0.00		0.00		0.00	
ANALYST REL DEV, %	16.74		6.39		7.84		5.98	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	7	6	7	6	6	6	5	5
TRUE CONC (C) UG/ML	0.03	0.02	0.03	0.02	0.03	0.02	0.03	0.02
MEAN RECOVERY (X)	0.03	0.02	0.02	0.02	0.03	0.02	0.03	0.02
% RECOVERY	81.70	88.89	74.11	86.81	91.67	87.50	90.00	86.67
OVERALL STD DEV (S)	0.01	0.00	0.01	0.00	0.00	0.01	0.00	0.00
OVERALL REL STD DEV, %	31.96	17.46	41.94	22.80	15.50	27.60	12.37	17.47
SINGLE STD DEV, (SR)	0.00		0.00		0.00		0.00	
ANALYST REL DEV, %	15.35		8.03		6.32		6.05	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 39. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

STATISTICAL SUMMARY FOR CADMIUM, CD ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	8	8	8	8	7	7	7	7
TRUE CONC (C) UG/ML	2.00	1.60	2.00	1.60	2.00	1.60	2.00	1.60
MEAN RECOVERY (X)	2.00	1.59	1.98	1.57	2.01	1.59	1.99	1.60
% RECOVERY	99.97	99.61	98.87	97.89	100.71	99.64	99.57	100.27
OVERALL STD DEV (S)	0.04	0.05	0.06	0.04	0.07	0.05	0.05	0.05
OVERALL REL STD DEV, %	2.17	3.39	3.04	2.60	3.56	3.32	2.42	2.98
SINGLE STD DEV, (SR)	0.03		0.03		0.04		0.04	
ANALYST REL DEV, %	1.92		1.82		2.48		2.28	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	9	8	8	8	7	7	7	7
TRUE CONC (C) UG/ML	0.30	0.60	0.80	0.60	0.80	0.60	0.80	0.60
MEAN RECOVERY (X)	0.80	0.59	0.79	0.59	0.78	0.58	0.79	0.59
% RECOVERY	99.86	98.33	98.59	98.12	97.68	96.90	99.11	99.05
OVERALL STD DEV (S)	0.03	0.02	0.03	0.03	0.04	0.03	0.03	0.02
OVERALL REL STD DEV, %	3.33	3.51	3.68	4.84	4.58	4.70	3.76	2.54
SINGLE STD DEV, (SR)	0.01		0.01		0.01		0.02	
ANALYST REL DEV, %	1.76		1.45		1.59		2.39	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	8	8	8	8	7	6	7	6
TRUE CONC (C) UG/ML	0.06	0.04	0.06	0.04	0.06	0.04	0.06	0.04
MEAN RECOVERY (X)	0.06	0.04	0.06	0.04	0.06	0.04	0.06	0.04
% RECOVERY	102.71	103.75	97.29	96.56	96.19	98.75	101.67	96.25
OVERALL STD DEV (S)	0.00	0.00	0.00	0.00	0.01	0.00	0.01	0.01
OVERALL REL STD DEV, %	4.33	8.54	6.91	6.63	16.67	7.29	12.41	15.30
SINGLE STD DEV, (SR)	0.00		0.00		0.00		0.00	
ANALYST REL DEV, %	4.11		4.53		6.74		3.48	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	7	7	7	5	6	6	6	5
TRUE CONC (C) UG/ML	0.03	0.02	0.03	0.02	0.03	0.02	0.03	0.02
MEAN RECOVERY (X)	0.03	0.03	0.03	0.02	0.03	0.02	0.03	0.02
% RECOVERY	105.80	111.31	103.12	92.50	98.44	95.14	104.69	100.83
OVERALL STD DEV (S)	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.00
OVERALL REL STD DEV, %	14.41	19.77	11.61	5.87	7.97	16.48	9.76	15.01
SINGLE STD DEV, (SR)	0.00		0.00		0.00		0.00	
ANALYST REL DEV, %	3.71		7.77		7.11		7.35	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 40. STUDY 38, SW 846 METHOD 3010, TRACE METALS, AA FLAME

STATISTICAL SUMMARY FOR CALCIUM, CA ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	9	9	6	6	6	6	7	7
TRUE CONC (C) UG/ML	120.00	90.00	120.00	90.00	120.00	90.00	120.00	90.00
MEAN RECOVERY (X)	121.70	91.20	114.65	87.93	121.83	95.85	111.86	83.89
% RECOVERY	101.42	101.33	95.54	97.70	101.53	106.50	93.21	93.21
OVERALL STD DEV (S)	19.93	18.68	20.64	11.04	10.53	12.58	19.06	14.40
OVERALL REL STD DEV, %	16.38	20.48	18.00	12.55	8.65	13.12	17.08	17.16
SINGLE STD DEV, (SR)	7.37		9.48		3.00		4.57	
ANALYST REL DEV, %	6.92		9.36		2.76		4.67	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	9	9	6	6	6	6	7	7
TRUE CONC (C) UG/ML	60.00	45.00	60.00	45.00	60.00	45.00	60.00	45.00
MEAN RECOVERY (X)	59.82	48.39	55.85	39.78	62.30	47.83	55.70	41.11
% RECOVERY	99.70	107.53	93.08	88.41	103.83	106.30	92.83	91.37
OVERALL STD DEV (S)	9.27	8.39	8.43	7.15	8.55	4.98	8.56	4.38
OVERALL REL STD DEV, %	15.49	17.33	15.09	17.97	13.72	10.40	15.37	10.56
SINGLE STD DEV, (SR)	6.21		5.36		4.55		3.25	
ANALYST REL DEV, %	11.48		10.58		8.27		6.71	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	9	9	6	6	5	6	7	7
TRUE CONC (C) UG/ML	24.00	18.00	24.00	18.00	24.00	18.00	24.00	18.00
MEAN RECOVERY (X)	25.44	20.00	21.43	15.65	24.10	18.67	23.13	18.84
% RECOVERY	105.99	111.11	89.31	86.94	100.42	103.70	96.37	104.68
OVERALL STD DEV (S)	2.09	2.32	1.60	0.61	0.77	2.18	2.65	2.49
OVERALL REL STD DEV, %	8.22	14.11	7.48	3.89	3.20	11.69	11.46	13.22
SINGLE STD DEV, (SR)	1.15		1.17		1.50		3.08	
ANALYST REL DEV, %	5.07		6.29		7.04		14.47	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	9	9	6	6	6	5	7	6
TRUE CONC (C) UG/ML	12.00	9.00	12.00	9.00	12.00	9.00	12.00	9.00
MEAN RECOVERY (X)	12.92	9.00	9.43	5.61	13.37	8.58	12.19	8.41
% RECOVERY	107.69	100.01	78.60	62.33	111.39	95.36	101.61	93.41
OVERALL STD DEV (S)	1.87	0.82	1.99	2.53	2.87	0.82	3.05	1.13
OVERALL REL STD DEV, %	14.48	9.13	21.07	45.13	21.48	9.56	24.99	13.40
SINGLE STD DEV, (SR)	1.46		0.88		1.62		0.74	
ANALYST REL DEV, %	13.36		11.66		14.75		7.16	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 41. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

STATISTICAL SUMMARY FOR CHROMIUM, CR ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	8	7	5	5	6	6	7	7
TRUE CONC (C) UG/ML	10.00	8.00	10.00	8.00	10.00	8.00	10.00	8.00
MEAN RECOVERY (X)	11.09	8.46	11.15	8.70	11.40	8.99	11.03	8.62
% RECOVERY	110.86	105.77	111.54	108.80	114.00	112.44	110.29	107.71
OVERALL STD DEV (S)	1.14	0.76	1.07	0.62	0.88	0.77	1.38	0.88
OVERALL REL STD DEV, %	10.26	9.03	9.57	7.11	7.73	8.54	12.55	10.24
SINGLE STD DEV, (SR)	0.35		0.38		0.36		0.42	
ANALYST REL DEV, %	3.63		3.46		3.55		4.23	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	8	8	5	5	6	6	7	7
TRUE CONC (C) UG/ML	5.00	4.00	5.00	4.00	5.00	4.00	5.00	4.00
MEAN RECOVERY (X)	5.31	4.34	5.33	4.25	5.48	4.25	5.28	4.19
% RECOVERY	106.25	108.47	106.56	106.20	109.67	106.21	105.69	104.75
OVERALL STD DEV (S)	0.43	0.39	0.52	0.41	0.32	0.35	0.51	0.35
OVERALL REL STD DEV, %	8.19	9.00	9.81	9.69	5.90	8.34	9.61	8.35
SINGLE STD DEV, (SR)	0.08		0.13		0.14		0.13	
ANALYST REL DEV, %	1.76		2.82		2.87		2.69	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	8	8	5	5	6	6	6	7
TRUE CONC (C) UG/ML	0.60	0.50	0.60	0.50	0.60	0.50	0.60	0.50
MEAN RECOVERY (X)	0.61	0.52	0.65	0.54	0.60	0.51	0.66	0.50
% RECOVERY	101.04	104.75	108.67	108.40	99.44	101.33	110.00	100.86
OVERALL STD DEV (S)	0.06	0.06	0.06	0.07	0.06	0.08	0.09	0.15
OVERALL REL STD DEV, %	10.24	11.27	9.42	12.20	10.26	15.07	14.12	29.28
SINGLE STD DEV, (SR)	0.07		0.02		0.05		0.02	
ANALYST REL DEV, %	12.07		2.78		8.73		4.14	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	8	8	5	5	6	6	7	6
TRUE CONC (C) UG/ML	0.30	0.24	0.30	0.24	0.30	0.24	0.30	0.24
MEAN RECOVERY (X)	0.30	0.24	0.31	0.23	0.32	0.27	0.32	0.31
% RECOVERY	101.67	100.52	102.00	95.00	107.22	113.89	106.19	127.78
OVERALL STD DEV (S)	0.04	0.03	0.03	0.04	0.05	0.06	0.14	0.06
OVERALL REL STD DEV, %	13.46	14.26	9.69	19.22	17.07	22.15	44.96	20.38
SINGLE STD DEV, (SR)	0.02		0.04		0.02		0.02	
ANALYST REL DEV, %	6.48		13.29		5.51		7.70	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 42. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

STATISTICAL SUMMARY FOR COBALT, CO ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	6	5	7	7	6	7	5	4
TRUE CONC (C) UG/ML	15.00	12.50	15.00	12.50	15.00	12.50	15.00	12.50
MEAN RECOVERY (X)	14.78	12.06	14.80	12.50	14.53	12.70	15.30	12.05
% RECOVERY	98.56	96.48	98.67	100.00	96.89	101.60	102.00	96.40
OVERALL STD DEV (S)	1.13	0.41	1.05	1.24	0.66	1.21	1.49	0.13
OVERALL REL STD DEV, %	7.62	3.40	7.11	9.92	4.54	9.51	9.77	1.07
SINGLE STD DEV, (SR)	0.38		0.20		0.38		0.57	
ANALYST REL DEV, %	2.85		1.50		2.76		4.17	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	6	6	7	7	7	7	4	4
TRUE CONC (C) UG/ML	7.50	6.00	7.50	6.00	7.50	6.00	7.50	6.00
MEAN RECOVERY (X)	7.21	5.86	7.21	5.85	7.62	6.10	7.23	5.81
% RECOVERY	96.20	97.69	96.10	97.57	101.66	101.62	96.47	96.79
OVERALL STD DEV (S)	0.33	0.24	0.72	0.47	0.53	0.71	0.10	0.08
OVERALL REL STD DEV, %	4.63	4.14	10.01	8.06	6.98	11.69	1.36	1.37
SINGLE STD DEV, (SR)	0.14		0.20		0.32		0.04	
ANALYST REL DEV, %	2.15		3.12		4.66		0.58	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	5	5	7	7	7	7	5	5
TRUE CONC (C) UG/ML	3.50	2.50	3.50	2.50	3.50	2.50	3.50	2.50
MEAN RECOVERY (X)	3.44	2.45	3.37	2.40	3.76	2.80	3.40	2.47
% RECOVERY	98.17	98.16	96.20	96.06	107.35	111.83	97.14	98.64
OVERALL STD DEV (S)	0.12	0.10	0.26	0.22	0.35	0.38	0.11	0.13
OVERALL REL STD DEV, %	3.40	4.06	7.63	9.26	9.35	13.43	3.31	5.26
SINGLE STD DEV, (SR)	0.38		0.10		0.25		0.05	
ANALYST REL DEV, %	1.50		3.41		7.78		1.68	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	6	6	7	7	7	7	5	5
TRUE CONC (C) UG/ML	1.50	1.00	1.50	1.00	1.50	1.00	1.50	1.00
MEAN RECOVERY (X)	1.50	0.98	1.47	0.98	1.65	1.16	1.50	0.96
% RECOVERY	99.89	98.33	97.81	98.00	110.00	116.29	99.73	96.40
OVERALL STD DEV (S)	0.36	0.05	0.14	0.11	0.22	0.39	0.08	0.04
OVERALL REL STD DEV, %	3.74	5.17	9.64	11.29	13.53	33.37	5.23	3.85
SINGLE STD DEV, (SR)	0.02		0.03		0.15		0.04	
ANALYST REL DEV, %	1.47		2.56		10.66		3.29	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 43. STUDY 38, SW 846 METHOD 3010, TRACE METALS, AA FLAME

STATISTICAL SUMMARY FOR COPPER , CU ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	7	7	7	7	8	8	7	7
TRUE CONC (C) UG/ML	5.00	4.00	5.00	4.00	5.00	4.00	5.00	4.00
MEAN RECOVERY (X)	5.02	3.97	4.95	3.94	4.98	4.01	4.92	4.00
% RECOVERY	100.49	99.14	99.06	98.61	99.55	100.31	98.46	100.11
OVERALL STD DEV (S)	0.14	0.16	0.16	0.12	0.21	0.13	0.23	0.20
OVERALL REL STD DEV, %	2.79	3.92	3.31	3.16	4.20	3.19	4.75	4.93
SINGLE STD DEV, (SR)	0.09		0.10		0.08		0.06	
ANALYST REL DEV, %	1.97		2.28		1.72		1.38	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	7	7	6	7	8	8	7	7
TRUE CONC (C) UG/ML	2.00	1.50	2.00	1.50	2.00	1.50	2.00	1.50
MEAN RECOVERY (X)	2.00	1.49	1.98	1.49	2.00	1.52	1.99	1.51
% RECOVERY	100.14	99.62	99.00	99.05	100.06	101.25	99.64	100.95
OVERALL STD DEV (S)	0.07	0.05	0.03	0.07	0.05	0.04	0.07	0.03
OVERALL REL STD DEV, %	3.27	3.09	1.53	4.77	2.72	2.91	3.35	2.25
SINGLE STD DEV, (SR)	0.02		0.04		0.02		0.03	
ANALYST REL DEV, %	1.13		2.34		1.35		1.73	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	7	7	7	7	7	7	7	7
TRUE CONC (C) UG/ML	0.25	0.20	0.25	0.20	0.25	0.20	0.25	0.20
MEAN RECOVERY (X)	0.26	0.20	0.25	0.20	0.26	0.20	0.25	0.20
% RECOVERY	102.29	101.43	100.57	100.00	102.86	101.43	99.43	100.71
OVERALL STD DEV (S)	0.01	0.01	0.02	0.02	0.01	0.01	0.01	0.01
OVERALL REL STD DEV, %	4.43	6.80	7.76	10.00	4.33	6.80	4.30	7.27
SINGLE STD DEV, (SR)	0.01		0.00		0.01		0.00	
ANALYST REL DEV, %	4.95		1.18		3.00		1.53	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	6	7	6	5	7	7	7	7
TRUE CONC (C) UG/ML	0.15	0.10	0.15	0.10	0.15	0.10	0.15	0.10
MEAN RECOVERY (X)	0.15	0.11	0.15	0.10	0.15	0.10	0.15	0.10
% RECOVERY	100.00	105.43	97.78	99.80	102.86	97.29	101.90	103.71
OVERALL STD DEV (S)	0.00	0.01	0.01	0.00	0.01	0.02	0.01	0.01
OVERALL REL STD DEV, %	UND	7.72	3.52	0.44	7.35	20.53	7.28	12.65
SINGLE STD DEV, (SR)	0.01		0.00		0.01		0.01	
ANALYST REL DEV, %	4.52		2.99		11.29		3.93	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 44. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

STATISTICAL SUMMARY FOR IRON, FE ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	7	6	7	7	6	6	8	8
TRUE CONC (C) UG/ML	5.00	4.00	5.00	4.00	5.00	4.00	5.00	4.00
MEAN RECOVERY (X)	4.60	3.65	4.74	3.77	4.85	3.81	5.10	4.27
% RECOVERY	91.97	91.29	94.77	94.36	97.00	95.37	102.07	106.66
OVERALL STD DEV (S)	0.44	0.30	0.38	0.50	0.16	0.20	0.46	0.59
OVERALL REL STD DEV, %	9.67	8.24	8.11	13.24	3.30	5.24	9.08	13.75
SINGLE STD DEV, (SR)	0.17		0.12		0.13		0.18	
ANALYST REL DEV, %	4.05		2.79		2.95		3.94	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	7	7	7	7	7	7	8	8
TRUE CONC (C) UG/ML	2.50	2.00	2.50	2.00	2.50	2.00	2.50	2.00
MEAN RECOVERY (X)	2.34	1.87	2.25	1.85	2.41	1.96	2.55	2.06
% RECOVERY	93.77	93.36	89.89	92.36	96.34	98.14	102.00	103.12
OVERALL STD DEV (S)	0.22	0.13	0.17	0.16	0.15	0.15	0.20	0.13
OVERALL REL STD DEV, %	9.59	6.75	7.39	8.49	6.26	7.87	7.90	6.18
SINGLE STD DEV, (SR)	0.38		0.16		0.04		0.09	
ANALYST REL DEV, %	3.85		8.05		1.72		4.09	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	7	7	7	7	7	7	8	6
TRUE CONC (C) UG/ML	0.40	0.30	0.40	0.30	0.40	0.30	0.40	0.30
MEAN RECOVERY (X)	0.37	0.30	0.22	0.19	0.36	0.24	0.28	0.21
% RECOVERY	92.50	98.57	56.25	63.19	91.07	79.76	70.31	70.56
OVERALL STD DEV (S)	0.05	0.04	0.11	0.11	0.13	0.09	0.15	0.07
OVERALL REL STD DEV, %	12.77	14.33	50.17	58.76	35.89	37.97	55.00	32.24
SINGLE STD DEV, (SR)	0.06		0.07		0.08		0.04	
ANALYST REL DEV, %	16.77		33.29		24.99		17.56	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	7	6	6	4	6	4	7	7
TRUE CONC (C) UG/ML	0.20	0.15	0.20	0.15	0.20	0.15	0.20	0.15
MEAN RECOVERY (X)	0.19	0.14	0.14	0.10	0.20	0.11	0.21	0.16
% RECOVERY	95.71	91.11	69.33	65.00	100.83	76.67	103.86	105.43
OVERALL STD DEV (S)	0.02	0.01	0.06	0.03	0.04	0.01	0.15	0.14
OVERALL REL STD DEV, %	9.74	10.00	45.90	34.91	19.18	8.70	73.74	89.87
SINGLE STD DEV, (SR)	0.01		0.01		0.03		0.07	
ANALYST REL DEV, %	5.04		4.89		20.79		39.51	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 45. STUDY 18,SW 846 METHOD 3010,TRACE METALS,AA FLAME

STATISTICAL SUMMARY FOR LEAD, PB ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	7	7	8	8	8	8	7	6
TRUE CONC (C) UG/ML	20.00	16.00	20.00	16.00	20.00	16.00	20.00	16.00
MEAN RECOVERY (X)	20.60	16.34	19.97	16.34	20.62	17.00	19.26	15.95
% RECOVERY	103.00	102.14	99.87	102.11	103.12	106.25	96.29	99.69
OVERALL STD DEV (S)	0.41	0.65	1.16	0.82	1.06	1.24	1.92	0.59
OVERALL REL STD DEV, %	2.00	3.96	5.80	4.99	5.12	7.27	9.99	3.67
SINGLE STD DEV, (SR)	0.58		0.70		0.58		1.00	
ANALYST REL DEV, %	3.15		3.56		3.08		5.66	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	6	7	8	8	7	8	7	7
TRUE CONC (C) UG/ML	8.00	6.00	8.00	6.00	8.00	6.00	8.00	6.00
MEAN RECOVERY (X)	8.27	6.19	8.12	6.22	8.27	6.16	7.92	5.88
% RECOVERY	103.35	103.17	101.55	103.73	103.36	102.65	99.00	98.07
OVERALL STD DEV (S)	0.22	0.12	0.40	0.27	0.35	0.29	0.37	0.45
OVERALL REL STD DEV, %	2.64	1.99	4.88	4.37	4.27	4.68	4.62	7.69
SINGLE STD DEV, (SR)	0.07		0.17		0.20		0.27	
ANALYST REL DEV, %	0.91		2.37		2.80		3.96	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	8	7	9	8	8	8	6	6
TRUE CONC (C) UG/ML	1.20	0.80	1.20	0.80	1.20	0.80	1.20	0.80
MEAN RECOVERY (X)	1.20	0.80	1.28	0.85	1.21	0.75	1.24	0.81
% RECOVERY	100.31	100.36	106.98	106.09	100.73	93.75	103.33	100.83
OVERALL STD DEV (S)	0.11	0.10	0.09	0.04	0.12	0.12	0.04	0.03
OVERALL REL STD DEV, %	9.50	12.37	6.93	4.81	9.69	15.71	3.14	3.65
SINGLE STD DEV, (SR)	0.05		0.08		0.04		0.03	
ANALYST REL DEV, %	4.69		7.56		4.47		2.61	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	8	6	8	6	8	7	6	7
TRUE CONC (C) UG/ML	0.60	0.40	0.60	0.40	0.60	0.40	0.60	0.40
MEAN RECOVERY (X)	0.59	0.41	0.65	0.49	0.56	0.44	0.61	0.45
% RECOVERY	98.54	103.33	107.92	122.08	94.17	109.29	101.39	111.43
OVERALL STD DEV (S)	0.09	0.07	0.04	0.05	0.10	0.13	0.04	0.04
OVERALL REL STD DEV, %	14.65	15.88	5.64	10.48	17.47	28.72	6.77	9.94
SINGLE STD DEV, (SR)	0.05		0.02		0.15		0.03	
ANALYST REL DEV, %	10.63		4.22		29.65		6.50	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 46. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

STATISTICAL SUMMARY FOR MAGNESIUM, MG ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	7	7	6	6	6	6	6	7
TRUE CONC (C) UG/ML	40.00	30.00	40.00	30.00	40.00	30.00	40.00	30.00
MEAN RECOVERY (X)	41.27	30.51	40.62	30.07	41.27	31.12	40.37	30.44
% RECOVERY	103.18	101.71	101.54	100.22	103.17	103.72	100.92	101.48
OVERALL STD DEV (S)	1.76	1.37	1.33	0.96	2.88	1.68	0.87	1.90
OVERALL REL STD DEV, %	4.27	4.49	3.28	3.19	6.99	5.39	2.16	6.26
SINGLE STD DEV, (SR)	0.71		0.65		2.17		0.65	
ANALYST REL DEV, %	1.97		1.33		5.99		1.84	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	8	7	6	6	6	5	6	7
TRUE CONC (C) UG/ML	20.00	15.00	20.00	15.00	20.00	15.00	20.00	15.00
MEAN RECOVERY (X)	20.60	15.43	19.87	15.50	20.47	14.86	20.07	15.20
% RECOVERY	103.00	102.86	99.33	103.33	102.33	99.07	100.33	101.33
OVERALL STD DEV (S)	1.25	0.70	0.72	0.65	1.66	0.43	0.47	0.46
OVERALL REL STD DEV, %	6.05	4.53	3.61	4.20	8.12	2.88	2.35	3.04
SINGLE STD DEV, (SR)	0.42		0.24		1.55		0.26	
ANALYST REL DEV, %	2.32		1.33		8.75		1.45	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	6	6	6	6	6	6	7	7
TRUE CONC (C) UG/ML	7.30	5.00	7.00	5.00	7.00	5.00	7.00	5.00
MEAN RECOVERY (X)	7.12	5.14	6.81	4.87	7.07	4.98	7.01	5.04
% RECOVERY	101.74	102.73	97.33	97.37	101.35	99.57	100.18	100.83
OVERALL STD DEV (S)	0.13	0.06	0.30	0.33	0.47	0.36	0.19	0.20
OVERALL REL STD DEV, %	1.82	1.08	4.42	6.82	6.70	7.24	2.73	3.92
SINGLE STD DEV, (SR)	0.11		0.30		0.27		0.16	
ANALYST REL DEV, %	1.72		5.17		8.44		2.69	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	8	8	6	6	6	6	7	7
TRUE CONC (C) UG/ML	3.00	2.00	3.00	2.00	3.00	2.00	3.00	2.00
MEAN RECOVERY (X)	3.12	2.01	2.92	2.05	3.21	1.32	2.89	1.98
% RECOVERY	103.96	100.56	97.39	102.58	106.39	96.00	96.29	99.07
OVERALL STD DEV (S)	0.16	0.05	0.10	0.15	0.10	0.61	0.22	0.17
OVERALL REL STD DEV, %	5.09	2.32	3.29	7.45	3.21	32.03	7.65	8.58
SINGLE STD DEV, (SR)	0.13		0.10		0.38		0.14	
ANALYST REL DEV, %	5.16		4.14		14.66		5.92	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 47. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

STATISTICAL SUMMARY FOR MANGANESE, MN ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	7	7	6	6	7	7	7	6
TRUE CONC (C) UG/ML	3.00	2.50	3.00	2.50	3.00	2.50	3.00	2.50
MEAN RECOVERY (X)	3.08	2.50	2.99	2.44	2.99	2.50	3.04	2.58
% RECOVERY	102.81	100.00	99.83	97.80	99.81	99.83	101.43	103.40
OVERALL STD DEV (S)	0.04	0.08	0.15	0.13	0.09	0.09	0.10	0.04
OVERALL REL STD DEV, %	1.31	3.36	4.85	5.22	3.15	3.76	3.37	1.40
SINGLE STD DEV, (SR)	0.05		0.04		0.03		0.06	
ANALYST REL DEV, %	1.69		1.32		1.02		2.26	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	8	8	6	7	7	7	7	7
TRUE CONC (C) UG/ML	1.20	1.00	1.20	1.00	1.20	1.00	1.20	1.00
MEAN RECOVERY (X)	1.24	1.04	1.15	0.97	1.21	1.00	1.22	1.01
% RECOVERY	103.44	104.25	95.97	97.00	100.60	99.71	101.55	101.00
OVERALL STD DEV (S)	0.08	0.07	0.03	0.05	0.08	0.04	0.06	0.04
OVERALL REL STD DEV, %	6.19	6.42	2.71	5.42	6.46	4.47	4.79	3.52
SINGLE STD DEV, (SR)	0.04		0.02		0.03		0.02	
ANALYST REL DEV, %	3.13		1.67		2.48		2.09	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	7	8	7	7	7	7	7	7
TRUE CONC (C) UG/ML	0.15	0.10	0.15	0.10	0.15	0.10	0.15	0.10
MEAN RECOVERY (X)	0.15	0.11	0.14	0.08	0.15	0.10	0.15	0.11
% RECOVERY	101.90	109.50	90.48	80.43	100.95	102.57	102.86	110.00
OVERALL STD DEV (S)	0.01	0.02	0.02	0.01	0.02	0.02	0.01	0.01
OVERALL REL STD DEV, %	6.22	17.92	13.36	13.54	12.89	14.87	6.33	9.09
SINGLE STD DEV, (SR)	0.01		0.01		0.01		0.00	
ANALYST REL DEV, %	6.60		8.44		6.72		2.86	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	7	7	5	5	6	4	6	5
TRUE CONC (C) UG/ML	0.07	0.05	0.07	0.05	0.07	0.05	0.07	0.05
MEAN RECOVERY (X)	0.07	0.05	0.05	0.03	0.07	0.05	0.08	0.06
% RECOVERY	96.33	95.43	72.29	63.60	97.86	95.00	110.71	110.80
OVERALL STD DEV (S)	0.01	0.01	0.01	0.01	0.01	0.00	0.01	0.00
OVERALL REL STD DEV, %	22.01	23.14	17.26	42.33	16.61	5.01	8.23	5.20
SINGLE STD DEV, (SR)	0.01		0.00		0.01		0.00	
ANALYST REL DEV, %	12.50		10.68		14.30		3.61	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 48. STUDY 38, SW 846 METHOD 3010, TRACE METALS, AA FLAME

STATISTICAL SUMMARY FOR MOLYBDENUM, NO ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	6	6	7	7	7	7	8	8
TRUE CONC (C) UG/ML	44.80	33.60	44.80	33.60	44.80	33.60	44.80	33.60
MEAN RECOVERY (X)	47.80	38.67	46.57	33.88	47.17	38.79	45.60	38.65
% RECOVERY	119.50	128.89	116.43	112.81	117.93	115.95	114.00	115.50
OVERALL STD DEV (S)	6.09	6.28	7.05	3.75	6.47	3.08	7.73	5.64
OVERALL REL STD DEV, %	12.75	16.24	15.14	11.07	13.71	8.86	16.96	16.29
SINGLE STD DEV, (SR)	5.20		2.77		2.65		2.36	
ANALYST REL DEV, %	12.02		6.30		6.47		5.88	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	6	6	6	6	6	7	7	6
TRUE CONC (C) UG/ML	16.80	11.20	16.80	11.20	16.80	11.20	16.80	11.20
MEAN RECOVERY (X)	20.45	13.40	16.98	11.10	16.43	12.53	16.24	11.22
% RECOVERY	136.33	134.00	113.22	111.00	112.22	125.29	108.29	112.17
OVERALL STD DEV (S)	6.43	3.90	1.63	0.80	1.14	3.16	1.66	0.51
OVERALL REL STD DEV, %	31.43	29.08	9.59	7.18	6.77	25.23	10.19	4.56
SINGLE STD DEV, (SR)	1.31		0.63		0.51		0.45	
ANALYST REL DEV, %	10.72		4.51		3.46		3.28	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	6	6	7	6	7	6	8	8
TRUE CONC (C) UG/ML	1.68	1.12	1.68	1.12	1.68	1.12	1.68	1.12
MEAN RECOVERY (X)	1.73	1.26	1.70	1.16	1.97	1.24	1.72	1.17
% RECOVERY	115.11	126.00	113.62	115.67	131.33	124.33	115.00	117.25
OVERALL STD DEV (S)	0.51	0.24	0.14	0.09	0.24	0.09	0.19	0.13
OVERALL REL STD DEV, %	29.49	19.26	8.12	7.37	12.27	6.91	11.13	10.93
SINGLE STD DEV, (SR)	0.37		0.07		0.10		0.14	
ANALYST REL DEV, %	24.79		5.15		6.28		9.85	
LOW YOUDEN PAIR	7	9	7	8	7	9	7	8
NUMBER OF DATA POINTS	6	6	7	7	7	7	8	8
TRUE CONC (C) UG/ML	0.84	0.56	0.84	0.56	0.84	0.56	0.84	0.56
MEAN RECOVERY (X)	0.73	0.50	0.80	0.52	0.37	1.62	0.80	0.63
% RECOVERY	104.76	100.00	114.49	104.46	124.49	123.14	114.11	125.50
OVERALL STD DEV (S)	0.13	0.17	0.12	0.25	0.07	0.23	0.17	0.25
OVERALL REL STD DEV, %	18.21	34.08	14.72	48.74	7.54	37.52	21.49	39.97
SINGLE STD DEV, (SR)	0.06		0.20		0.20		0.09	
ANALYST REL DEV, %	9.36		29.83		26.43		12.68	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 49. STUDY 38, SW 846 METHOD 3010, TRACE METALS, AA FLAME

STATISTICAL SUMMARY FOR NICKEL, NI ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	8	7	9	9	7	7	7	7
TRUE CONC (C) UG/ML	5.00	4.00	5.00	4.00	5.00	4.00	5.00	4.00
MEAN RECOVERY (X)	4.91	3.93	4.87	3.88	5.00	4.03	5.01	4.06
% RECOVERY	98.25	98.21	97.33	97.00	99.94	100.75	100.29	101.43
OVERALL STD DEV (S)	0.23	0.17	0.24	0.24	0.18	0.27	0.24	0.20
OVERALL REL STD DEV, %	4.68	4.29	5.02	6.16	3.61	6.76	4.76	4.82
SINGLE STD DEV, (SR)	0.05		0.07		0.17		0.09	
ANALYST REL DEV, %	1.21		1.65		3.68		1.33	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	8	7	9	9	7	7	7	7
TRUE CONC (C) UG/ML	2.00	1.50	2.00	1.50	2.00	1.50	2.00	1.50
MEAN RECOVERY (X)	1.96	1.48	1.96	1.46	2.02	1.52	2.01	1.51
% RECOVERY	97.81	98.57	97.89	97.41	101.14	101.14	100.29	100.38
OVERALL STD DEV (S)	0.04	0.04	0.06	0.07	0.09	0.11	0.07	0.05
OVERALL REL STD DEV, %	1.95	2.93	3.09	4.95	4.42	7.42	3.46	3.09
SINGLE STD DEV, (SR)	0.02		0.05		0.07		0.05	
ANALYST REL DEV, %	1.34		2.76		4.22		3.00	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	8	3	9	7	7	7	7	7
TRUE CONC (C) UG/ML	0.40	0.30	0.40	0.30	0.40	0.30	0.40	0.30
MEAN RECOVERY (X)	0.37	0.28	0.38	0.29	0.41	0.27	0.40	0.29
% RECOVERY	93.12	94.17	95.28	95.24	102.86	90.00	100.00	96.67
OVERALL STD DEV (S)	0.06	0.03	0.02	0.01	0.04	0.07	0.04	0.03
OVERALL REL STD DEV, %	15.70	9.02	6.04	2.75	8.92	26.10	10.99	9.55
SINGLE STD DEV, (SR)	0.03		0.01		0.04		0.05	
ANALYST REL DEV, %	7.66		4.39		12.48		13.18	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	8	8	9	9	7	7	7	7
TRUE CONC (C) UG/ML	0.20	0.15	0.20	0.15	0.20	0.15	0.20	0.15
MEAN RECOVERY (X)	0.16	0.14	0.19	0.13	0.19	0.15	0.20	0.16
% RECOVERY	82.37	96.67	94.44	88.15	95.00	98.29	101.43	108.57
OVERALL STD DEV (S)	0.05	0.02	0.02	0.03	0.05	0.06	0.04	0.04
OVERALL REL STD DEV, %	30.35	13.79	11.06	25.86	24.87	40.77	19.03	23.44
SINGLE STD DEV, (SR)	0.02		0.02		0.03		0.01	
ANALYST REL DEV, %	14.36		10.79		16.48		4.47	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 50. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

STATISTICAL SUMMARY FOR POTASSIUM, K ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	7	7	6	6	7	7	6	6
TRUE CONC (C) UG/ML	30.00	24.00	30.00	24.00	30.00	24.00	30.00	24.00
MEAN RECOVERY (X)	28.77	22.29	28.30	22.47	27.89	22.47	28.57	23.03
% RECOVERY	95.90	92.86	94.33	93.61	92.95	93.63	95.22	95.97
OVERALL STD DEV (S)	1.57	1.62	2.24	1.18	4.93	2.85	2.34	1.12
OVERALL REL STD DEV, %	5.46	7.26	7.91	5.24	17.69	12.70	8.20	4.86
SINGLE STD DEV, (SR)	0.39		0.92		1.62		1.21	
ANALYST REL DEV, %	1.54		3.62		6.42		4.70	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	7	7	6	6	7	7	6	7
TRUE CONC (C) UG/ML	15.00	12.00	15.00	12.00	15.00	12.00	15.00	12.00
MEAN RECOVERY (X)	14.04	10.67	14.65	11.27	14.29	11.41	15.25	11.01
% RECOVERY	93.62	88.92	97.67	93.89	95.24	95.12	101.67	91.73
OVERALL STD DEV (S)	1.58	1.05	0.90	0.77	0.98	0.63	1.13	1.80
OVERALL REL STD DEV, %	11.23	9.88	6.18	6.84	6.88	5.53	7.43	16.39
SINGLE STD DEV, (SR)	0.71		0.34		0.86		0.89	
ANALYST REL DEV, %	5.75		2.59		6.60		6.77	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	7	7	6	5	6	7	7	7
TRUE CONC (C) UG/ML	6.00	4.80	6.00	4.80	6.00	4.80	6.00	4.80
MEAN RECOVERY (X)	5.76	4.61	5.65	4.50	5.88	4.79	5.64	4.31
% RECOVERY	95.98	96.07	94.14	93.67	98.00	99.85	94.05	89.79
OVERALL STD DEV (S)	0.45	0.36	0.31	0.26	0.27	0.85	0.65	0.87
OVERALL REL STD DEV, %	7.85	7.91	5.54	5.71	4.68	17.77	11.53	20.08
SINGLE STD DEV, (SR)	0.42		0.31		0.47		0.86	
ANALYST REL DEV, %	8.05		6.15		8.88		9.28	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	7	7	5	6	5	6	7	7
TRUE CONC (C) UG/ML	3.00	2.40	3.00	2.40	3.00	2.40	3.00	2.40
MEAN RECOVERY (X)	2.69	2.17	2.82	2.18	2.76	2.40	3.29	2.87
% RECOVERY	89.62	90.48	94.07	90.83	92.07	99.93	109.57	119.64
OVERALL STD DEV (S)	0.27	0.23	0.08	0.27	0.19	0.48	0.42	0.78
OVERALL REL STD DEV, %	10.20	10.74	2.88	12.18	7.01	19.87	12.76	27.08
SINGLE STD DEV, (SR)	0.14		0.12		0.35		0.52	
ANALYST REL DEV, %	5.86		4.92		13.62		16.86	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 51. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

STATISTICAL SUMMARY FOR SILVER, AG ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	9	9	7	7	9	9	4	4
TRUE CONC (C) UG/ML	4.00	3.00	4.00	3.00	4.00	3.00	4.00	3.00
MEAN RECOVERY (X)	0.81	0.92	0.53	0.49	0.89	1.07	0.08	0.07
% RECOVERY	20.36	30.56	13.18	16.48	22.31	35.74	1.96	2.38
OVERALL STD DEV (S)	0.38	0.49	0.28	0.31	0.55	0.66	0.02	0.02
OVERALL REL STD DEV, %	46.75	53.49	52.36	62.06	61.51	61.97	24.47	25.39
SINGLE STD DEV, (SR)	0.36		0.11		0.30		0.01	
ANALYST REL DEV, %	41.98		21.53		30.17		16.24	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	9	9	7	7	9	9	4	6
TRUE CONC (C) UG/ML	1.50	1.00	1.50	1.00	1.50	1.00	1.50	1.00
MEAN RECOVERY (X)	0.81	0.69	0.33	0.31	0.56	0.55	0.05	0.30
% RECOVERY	54.15	69.00	22.29	31.14	37.63	55.44	3.62	29.87
OVERALL STD DEV (S)	0.56	0.30	0.22	0.21	0.41	0.22	0.02	0.29
OVERALL REL STD DEV, %	69.02	43.13	66.57	68.40	71.83	39.55	36.82	98.41
SINGLE STD DEV, (SR)	0.21		0.08		0.21		0.14	
ANALYST REL DEV, %	27.40		23.34		37.32		81.55	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	9	9	7	7	8	9	6	6
TRUE CONC (C) UG/ML	0.15	0.10	0.15	0.10	0.15	0.10	0.15	0.10
MEAN RECOVERY (X)	0.14	0.10	0.10	0.08	0.12	0.09	0.07	0.06
% RECOVERY	95.56	97.78	66.10	81.14	80.83	91.00	45.00	65.00
OVERALL STD DEV (S)	0.02	0.01	0.04	0.02	0.01	0.02	0.05	0.04
OVERALL REL STD DEV, %	13.95	12.50	44.71	24.44	6.38	20.01	80.82	69.19
SINGLE STD DEV, (SR)	0.01		0.02		0.01		0.01	
ANALYST REL DEV, %	6.63		20.51		10.14		12.70	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	9	9	7	7	8	9	5	6
TRUE CONC (C) UG/ML	0.07	0.05	0.07	0.05	0.07	0.05	0.07	0.05
MEAN RECOVERY (X)	0.08	0.06	0.07	0.06	0.06	0.06	0.04	0.05
% RECOVERY	108.89	119.33	102.86	123.14	90.58	114.22	54.57	109.00
OVERALL STD DEV (S)	0.02	0.02	0.02	0.02	0.02	0.02	0.01	0.04
OVERALL REL STD DEV, %	20.50	33.84	21.41	40.17	31.02	36.36	23.88	75.25
SINGLE STD DEV, (SR)	0.01		0.01		0.01		0.01	
ANALYST REL DEV, %	9.01		14.28		14.19		28.89	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 52. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

STATISTICAL SUMMARY FOR SODIUM, NA ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	9	8	6	6	5	5	4	5
TRUE CONC (C) UG/ML	600.00	480.00	600.30	480.00	600.00	480.00	600.00	480.00
MEAN RECOVERY (X)	619.78	482.75	583.83	480.83	610.60	469.60	594.25	487.20
% RECOVERY	103.30	100.57	97.31	100.17	101.77	97.83	99.04	101.50
OVERALL STD DEV (S)	45.94	29.14	22.23	29.76	39.28	49.92	6.08	30.11
OVERALL REL STD DEV, %	7.41	6.04	3.81	6.19	6.43	10.63	1.02	6.18
SINGLE STD DEV, (SR)	17.30		16.33		53.47		21.83	
ANALYST REL DEV, %	3.14		3.07		9.90		4.04	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	9	9	7	7	4	4	4	4
TRUE CONC (C) UG/ML	300.00	240.00	300.00	240.00	300.00	240.00	300.00	240.00
MEAN RECOVERY (X)	309.33	249.11	311.71	249.00	308.50	244.25	286.25	226.25
% RECOVERY	103.11	103.80	103.90	103.75	102.83	101.77	95.42	94.27
OVERALL STD DEV (S)	20.06	16.67	26.51	9.43	14.66	10.72	24.47	5.85
OVERALL REL STD DEV, %	6.48	6.69	8.50	3.79	4.75	4.39	8.55	2.59
SINGLE STD DEV, (SR)	9.65		12.66		6.78		16.84	
ANALYST REL DEV, %	3.46		4.52		2.44		6.57	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	9	8	7	7	5	5	4	4
TRUE CONC (C) UG/ML	120.00	96.00	120.00	96.00	120.00	96.00	120.00	96.00
MEAN RECOVERY (X)	120.78	99.15	121.14	97.36	119.80	97.82	116.50	95.75
% RECOVERY	100.65	103.28	100.95	101.41	99.83	101.90	97.08	99.74
OVERALL STD DEV (S)	4.61	5.93	5.09	6.04	9.73	3.65	5.45	5.59
OVERALL REL STD DEV, %	7.13	5.99	6.68	6.20	8.12	3.73	4.68	6.88
SINGLE STD DEV, (SR)	2.22		3.95		5.93		5.50	
ANALYST REL DEV, %	2.02		3.62		5.45		5.18	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	9	9	7	7	5	5	5	5
TRUE CONC (C) UG/ML	36.00	24.00	36.00	24.00	36.00	24.00	36.00	24.00
MEAN RECOVERY (X)	37.84	25.54	36.17	24.56	36.04	24.04	34.68	26.46
% RECOVERY	105.12	106.44	100.48	102.32	100.11	100.17	96.33	110.25
OVERALL STD DEV (S)	2.04	1.59	1.95	1.85	3.45	3.94	6.15	6.41
OVERALL REL STD DEV, %	5.40	6.24	5.40	7.54	9.57	16.40	17.73	24.24
SINGLE STD DEV, (SR)	0.80		0.48		2.01		4.96	
ANALYST REL DEV, %	2.52		1.58		6.69		16.23	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 53. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

STATISTICAL SUMMARY FOR THALLIUM, TI ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	9	8	6	6	8	8	9	9
TRUE CONC (C) UG/ML	20.00	16.00	20.00	16.00	20.00	16.00	20.00	16.00
MEAN RECOVERY (X)	20.54	16.55	20.65	16.40	20.52	16.32	20.47	16.61
% RECOVERY	102.72	103.44	103.25	102.50	102.62	102.03	102.33	103.82
OVERALL STD DEV (S)	0.51	0.50	0.61	0.28	0.52	0.33	0.62	0.64
OVERALL REL STD DEV, %	2.49	3.03	2.93	1.68	2.55	2.04	3.02	3.85
SINGLE STD DEV, (SR)	0.24		0.28		0.27		0.28	
ANALYST REL DEV, %	1.30		1.52		1.48		1.53	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	9	9	6	6	8	8	9	9
TRUE CONC (C) UG/ML	10.00	8.00	10.00	8.00	10.00	8.00	10.00	8.00
MEAN RECOVERY (X)	10.28	8.26	10.30	8.20	10.25	8.16	10.30	8.26
% RECOVERY	102.81	103.24	103.02	102.50	102.47	102.00	103.00	103.19
OVERALL STD DEV (S)	0.37	0.21	0.25	0.22	0.27	0.16	0.27	0.16
OVERALL REL STD DEV, %	3.57	2.53	2.43	2.70	2.63	1.91	2.66	1.96
SINGLE STD DEV, (SR)	0.15		0.16		0.19		0.17	
ANALYST REL DEV, %	1.53		1.77		2.07		1.85	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	9	9	6	6	8	7	9	9
TRUE CONC (C) UG/ML	1.20	1.00	1.20	1.00	1.20	1.00	1.20	1.00
MEAN RECOVERY (X)	1.24	0.99	1.19	1.02	1.20	0.97	1.24	1.02
% RECOVERY	103.32	99.00	98.89	102.17	100.10	97.43	103.24	102.11
OVERALL STD DEV (S)	0.08	0.07	0.10	0.06	0.06	0.07	0.07	0.09
OVERALL REL STD DEV, %	6.50	6.81	8.62	6.11	5.16	7.35	5.44	8.66
SINGLE STD DEV, (SR)	0.07		0.06		0.03		0.06	
ANALYST REL DEV, %	6.61		5.55		2.62		5.35	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	8	8	5	5	7	6	8	6
TRUE CONC (C) UG/ML	0.60	0.48	0.60	0.48	0.60	0.48	0.60	0.48
MEAN RECOVERY (X)	0.54	0.37	0.62	0.48	0.58	0.40	0.63	0.53
% RECOVERY	90.62	77.27	103.00	99.17	96.19	83.33	105.42	109.72
OVERALL STD DEV (S)	0.15	0.15	0.05	0.06	0.09	0.13	0.10	0.03
OVERALL REL STD DEV, %	27.22	39.92	7.71	12.12	16.21	32.90	16.25	6.75
SINGLE STD DEV, (SR)	0.03		0.03		0.05		0.04	
ANALYST REL DEV, %	5.47		5.04		10.78		7.38	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 54. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

STATISTICAL SUMMARY FOR VANADIUM, V ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	6	7	7	7	7	6	5	6
TRUE CONC (C) UG/ML	99.00	81.00	99.00	81.00	99.00	81.00	99.00	81.00
MEAN RECOVERY (X)	103.27	91.33	111.49	90.03	103.39	85.85	101.20	88.28
% RECOVERY	104.31	112.75	112.61	111.15	104.43	105.99	102.22	108.99
OVERALL STD DEV (S)	5.22	14.31	18.56	14.79	13.92	12.30	8.40	9.81
OVERALL REL STD DEV, %	5.05	15.66	16.64	16.43	13.47	14.33	8.34	11.11
SINGLE STD DEV, (SR)	10.61		3.35		2.46		0.84	
ANALYST REL DEV, %	10.91		3.33		2.60		0.89	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	7	6	7	6	7	7	6	5
TRUE CONC (C) UG/ML	45.00	36.00	45.00	36.00	45.00	36.00	45.00	36.00
MEAN RECOVERY (X)	47.16	37.03	47.97	37.63	47.10	38.33	48.23	37.32
% RECOVERY	104.79	102.37	106.60	104.54	104.67	106.47	107.19	103.67
OVERALL STD DEV (S)	3.27	1.89	5.01	2.46	4.06	5.33	4.71	2.18
OVERALL REL STD DEV, %	6.94	5.11	10.44	6.54	8.63	13.91	9.76	5.85
SINGLE STD DEV, (SR)	1.10		2.63		1.21		0.61	
ANALYST REL DEV, %	2.61		6.15		2.34		1.42	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	6	7	7	6	6	7	6	6
TRUE CONC (C) UG/ML	2.70	2.25	2.70	2.25	2.70	2.25	2.70	2.25
MEAN RECOVERY (X)	2.79	2.40	3.05	2.40	2.80	2.28	2.86	2.41
% RECOVERY	103.52	106.79	113.12	106.52	103.89	101.21	106.05	106.96
OVERALL STD DEV (S)	0.29	0.17	0.38	0.09	0.22	0.16	0.16	0.17
OVERALL REL STD DEV, %	10.38	7.05	12.32	3.81	7.68	7.18	5.65	7.11
SINGLE STD DEV, (SR)	0.24		0.28		0.08		0.05	
ANALYST REL DEV, %	9.37		10.42		2.97		2.08	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	7	7	7	6	7	7	6	6
TRUE CONC (C) UG/ML	1.35	1.08	1.35	1.08	1.35	1.08	1.35	1.08
MEAN RECOVERY (X)	1.41	1.12	1.39	1.12	1.33	0.99	1.39	1.09
% RECOVERY	104.23	103.31	102.65	104.01	98.20	91.30	103.09	101.23
OVERALL STD DEV (S)	0.14	0.13	0.11	0.10	0.18	0.18	0.16	0.23
OVERALL REL STD DEV, %	9.74	11.85	8.12	9.19	13.29	18.42	11.71	21.47
SINGLE STD DEV, (SR)	0.04		0.05		0.10		0.09	
ANALYST REL DEV, %	2.85		4.22		8.74		7.27	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

TABLE 55. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

STATISTICAL SUMMARY FOR ZINC, ZN ANALYSES BY WATER TYPE

	WATER 1		WATER 2		WATER 3		WATER 4	
HIGH YOUDEN PAIR	1	2	1	2	1	2	1	2
NUMBER OF DATA POINTS	5	5	5	5	6	6	8	8
TRUE CONC (C) UG/ML	1.00	0.80	1.00	0.80	1.00	0.80	1.00	0.80
MEAN RECOVERY (X)	1.00	0.79	0.89	0.72	1.00	0.80	0.93	0.74
% RECOVERY	100.00	99.25	89.00	90.25	99.83	99.79	92.62	93.12
OVERALL STD DEV (S)	0.04	0.03	0.04	0.07	0.08	0.09	0.05	0.03
OVERALL REL STD DEV, %	3.61	4.33	4.28	9.76	8.12	11.57	5.59	4.12
SINGLE STD DEV, (SR)	0.02		0.03		0.07		0.03	
ANALYST REL DEV, %	1.81		4.04		8.27		3.63	
HIGH-MEDIUM YOUDEN P	3	4	3	4	3	4	3	4
NUMBER OF DATA POINTS	5	5	5	5	6	6	8	8
TRUE CONC (C) UG/ML	0.50	0.40	0.50	0.40	0.50	0.40	0.50	0.40
MEAN RECOVERY (X)	0.49	0.39	0.42	0.36	0.48	0.40	0.49	0.40
% RECOVERY	97.60	98.50	83.60	89.00	96.33	100.42	98.25	99.37
OVERALL STD DEV (S)	0.02	0.02	0.08	0.06	0.04	0.04	0.04	0.03
OVERALL REL STD DEV, %	3.94	4.95	19.90	16.33	7.92	9.63	8.53	8.26
SINGLE STD DEV, (SR)	0.00		0.05		0.01		0.03	
ANALYST REL DEV, %	0.39		11.66		2.36		6.53	
LOW-MEDIUM YOUDEN PA	5	6	5	6	5	6	5	6
NUMBER OF DATA POINTS	5	5	5	5	6	6	8	8
TRUE CONC (C) UG/ML	0.15	0.10	0.15	0.10	0.15	0.10	0.15	0.10
MEAN RECOVERY (X)	0.16	0.10	0.09	0.03	0.18	0.12	0.15	0.10
% RECOVERY	106.67	101.80	58.27	35.00	118.89	116.17	100.83	96.75
OVERALL STD DEV (S)	0.02	0.01	0.01	0.03	0.05	0.06	0.03	0.03
OVERALL REL STD DEV, %	11.69	7.65	13.33	85.52	26.40	52.36	19.81	26.33
SINGLE STD DEV, (SR)	0.01		0.02		0.05		0.02	
ANALYST REL DEV, %	9.97		34.40		32.16		15.93	
LOW YOUDEN PAIR	7	8	7	8	7	8	7	8
NUMBER OF DATA POINTS	5	5	0	0	5	4	7	6
TRUE CONC (C) UG/ML	0.06	0.05	0.06	0.05	0.06	0.05	0.06	0.05
MEAN RECOVERY (X)	0.06	0.05	UND	UND	0.07	0.04	0.05	0.06
% RECOVERY	98.33	95.20	UND	UND	115.00	95.50	87.14	112.33
OVERALL STD DEV (S)	0.00	0.01	UND	UND	0.04	0.02	0.02	0.03
OVERALL REL STD DEV, %	8.22	11.56	UND	UND	61.34	51.26	38.77	48.39
SINGLE STD DEV, (SR)	0.00		UND		0.01		0.01	
ANALYST REL DEV, %	3.05		UND		14.52		15.26	

WATER LEGEND

1 - REAGENT WATER
2 - OHIO RIVER WATER
3 - MUNICIPAL SECONDARY EFFLUENT
4 - ELECTROPLATING EFFLUENT

APPENDIX D

EFFECT OF WATER TYPE

TABLE 56. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

EFFECT OF WATER TYPE ON ALUMINUM, AL ANALYSIS

** POINT ESTIMATES **

REAGENT WATER SLOPE:GAMMA(1) = 1.02603

WATER	INTERCEPT(WATER-REAGENT)	SLOPE(WATER-REAGENT)
WATER 1	-0.0460	-0.0052
WATER 2	-0.0261	0.0003
WATER 3	0.0380	-0.0117

** ANALYSIS OF VARIANCE **

SOURCE	DF	SUM OF SQUARES	MEAN SQUARE	F	PROB
REG(REAGENT)	1	129.32832	129.32832		
REG(WATER/REAGENT)	6	0.10620	0.01770	4.83	0.0002
ERROR	143	0.52369	0.00366		
TOTAL	150	129.95821			

** TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **

WATER	INTERCEPT(WATER-REAGENT) ESTIMATE	INTERCEPT(WATER-REAGENT) INTERVAL	SLOPE(WATER-REAGENT) ESTIMATE	SLOPE(WATER-REAGENT) INTERVAL
WATER 1	-0.0460	(-0.1585 , 0.0665)	-0.0052	(-0.0425 , 0.0320)
WATER 2	-0.0261	(-0.1871 , 0.0950)	0.0003	(-0.0401 , 0.0407)
WATER 3	0.0380	(-0.0895 , 0.1656)	-0.0117	(-0.0540 , 0.0305)

NOTE: IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

WATER 1 - OHIO RIVER WATER
WATER 2 - MUNICIPAL SECONDARY EFFLUENT
WATER 3 - ELECTROPLATING EFFLUENT

TABLE 57. STUDY 38, SW 846 METHOD 3010, TRACE METALS, AA FLAME

EFFECT OF WATER TYPE ON ANTIMONY, SB ANALYSIS

** POINT ESTIMATES **

REAGENT WATER SLOPE: GAMMA(1) = 1.07236

WATER	INTERCEPT(WATER-REAGENT)	SLOPE(WATER-REAGENT)
WATER 1	0.0379	-0.0122
WATER 2	0.1294	-0.0420
WATER 3	0.2261	-0.0728

** ANALYSIS OF VARIANCE **

SOURCE	DF	SUM OF SQUARES	MEAN SQUARE	F	PROB
REG(REAGENT)	1	393.76219	393.76219		
REG(WATER/REAGENT)	6	0.39660	0.06610	4.88	0.0001
ERROR	172	2.33087	0.01355		
TOTAL	179	396.48966			

** TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **

WATER	INTERCEPT(WATER-REAGENT) ESTIMATE	INTERCEPT(WATER-REAGENT) INTERVAL	SLOPE(WATER-REAGENT) ESTIMATE	SLOPE(WATER-REAGENT) INTERVAL
WATER 1	0.0379	(-0.0747 , 0.1505)	-0.0122	(-0.0559 , 0.0316)
WATER 2	0.1294	(0.0129 , 0.2458)	-0.0420	(-0.0870 , 0.0030)
WATER 3	0.2261	(0.1053 , 0.3468)	-0.0728	(-0.1203 , -0.0253)

NOTE: IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

WATER 1 - OHIO RIVER WATER
WATER 2 - MUNICIPAL SECONDARY EFFLUENT
WATER 3 - ELECTROPLATING EFFLUENT

TABLE 58. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

```
                    EFFECT OF WATER TYPE ON BARIUM, BA ANALYSIS

                              ** POINT ESTIMATES **

                 REAGENT WATER SLOPE:GAMMA(1) = 0.97612

WATER                    INTERCEPT(WATER-REAGENT)    SLOPE(WATER-REAGENT)

        WATER 1                   0.0527                   -0.0293
        WATER 2                   0.2108                   -0.3531
        WATER 3                   0.2679                   -0.6687


                            ** ANALYSIS OF VARIANCE **

                SOURCE           DF   SUM OF SQUARES  MEAN SQUARE    F     PROB

             REG(REAGENT)         1     131.85241     131.85241
             REG(WATER/REAGENT)   6      40.74571       6.79095    39.89 0.0000
             ERROR              179      30.47004       0.17022

             TOTAL              186     203.06816


** TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **

                           INTERCEPT(WATER-REAGENT)              SLOPE(WATER-REAGENT)
      WATER                ESTIMATE       INTERVAL            ESTIMATE       INTERVAL

             WATER 1        0.0527  ( -0.3541 ,   0.4595)  -0.0293  ( -0.2286 ,   0.1700)
             WATER 2        0.2108  ( -0.1506 ,   0.5722)  -0.3531  ( -0.5330 ,  -0.1733)
             WATER 3        0.2679  ( -0.1059 ,   0.6417)  -0.6687  ( -0.8541 ,  -0.4834)

NOTE:  IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN
       REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

       THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION
       AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

       WATER 1  -  OHIO RIVER WATER
       WATER 2  -  MUNICIPAL SECONDARY EFFLUENT
       WATER 3  -  ELECTROPLATING EFFLUENT
```

TABLE 59. STUDY 38, SW 846 METHOD 3010, TRACE METALS, AA FLAME

EFFECT OF WATER TYPE ON BERYLLIUM, BE ANALYSIS

** POINT ESTIMATES **

REAGENT WATER SLOPE:GAMMA(1) = 1.00735

WATER	INTERCEPT(WATER-REAGENT)	SLOPE(WATER-REAGENT)
WATER 1	0.0283	0.0018
WATER 2	0.0354	-0.0164
WATER 3	0.0103	-0.0046

** ANALYSIS OF VARIANCE **

SOURCE	DF	SUM OF SQUARES	MEAN SQUARE	F	PROB
REG(REAGENT)	1	302.36645	302.36645		
REG(WATER/REAGENT)	6	0.06481	0.01080	0.85	0.5315
ERROR	125	1.58260	0.01266		
TOTAL	132	304.01386			

** TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **

WATER	INTERCEPT(WATER-REAGENT) ESTIMATE	INTERCEPT(WATER-REAGENT) INTERVAL	SLOPE(WATER-REAGENT) ESTIMATE	SLOPE(WATER-REAGENT) INTERVAL
WATER 1	0.0283	(-0.0528 , 0.1094)	0.0018	(-0.0457 , 0.0493)
WATER 2	0.0354	(-0.0460 , 0.1168)	-0.0164	(-0.0619 , 0.0292)
WATER 3	0.0103	(-0.0782 , 0.0987)	-0.0046	(-0.0533 , 0.0440)

NOTE: IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

WATER 1 - OHIO RIVER WATER
WATER 2 - MUNICIPAL SECONDARY EFFLUENT
WATER 3 - ELECTROPLATING EFFLUENT

```
              TABLE 60.  STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

                           EFFECT OF WATER TYPE ON CADMIUM, CD ANALYSIS

                                   ** POINT ESTIMATES **

                       REAGENT WATER SLOPE:GAMMA(1) = 0.98973

   WATER                       INTERCEPT(WATER-REAGENT)     SLOPE(WATER-REAGENT)

        WATER 1                          -0.0166                  0.0144
        WATER 2                          -0.0109                  0.0060
        WATER 3                          -0.0026                  0.0053


                                  ** ANALYSIS OF VARIANCE **

                    SOURCE           DF   SUM OF SQUARES   MEAN SQUARE    F      PROB

             REG(REAGENT)             1      421.59767      421.59767
             REG(WATER/REAGENT)       6        0.03241        0.00540   0.95 0.4575
             ERROR                  168        0.95029        0.00566

             TOTAL                  175      422.58037


 ** TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **
                                  INTERCEPT(WATER-REAGENT)                SLOPE(WATER-REAGENT)
                                  ESTIMATE       INTERVAL               ESTIMATE      INTERVAL
   WATER

        WATER 1                   -0.0166  ( -0.0668 ,   0.0336)    0.0144  ( -0.0130 ,   0.0418)
        WATER 2                   -0.0109  ( -0.0587 ,   0.0369)    0.0060  ( -0.0208 ,   0.0328)
        WATER 3                   -0.0026  ( -0.0507 ,   0.0456)    0.0053  ( -0.0215 ,   0.0321)

NOTE:   IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN
        REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

        THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION
        AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

        WATER 1  -  OHIO RIVER WATER
        WATER 2  -  MUNICIPAL SECONDARY EFFLUENT
        WATER 3  -  ELECTROPLATING EFFLUENT
```

TABLE 61. STUDY 38,SW 846 METHOD 3013,TRACE METALS,AA FLAME

EFFECT OF WATER TYPE ON CALCIUM, CA ANALYSIS

** POINT ESTIMATES **

REAGENT WATER SLOPE:GAMMA(1) = 0.94323

WATER	INTERCEPT(WATER-REAGENT)	SLOPE(WATER-REAGENT)
WATER 1	-0.5921	0.1052
WATER 2	-0.0932	0.0198
WATER 3	-0.0951	0.0020

** ANALYSIS OF VARIANCE **

SOURCE	DF	SUM OF SQUARES	MEAN SQUARE	F	PROB
REG(REAGENT)	1	69.26354	69.26354		
REG(WATER/REAGENT)	6	0.89726	0.14954	9.23	0.0000
ERROR	147	2.38238	0.01621		
TOTAL	154	72.54318			

** TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **

	INTERCEPT(WATER-REAGENT)		SLOPE(WATER-REAGENT)	
WATER	ESTIMATE	INTERVAL	ESTIMATE	INTERVAL
WATER 1	-0.5921	(-1.0386 , -0.1457)	0.1052	(-0.0070 , 0.2173)
WATER 2	-0.0932	(-0.5334 , 0.3470)	0.0198	(-0.0911 , 0.1308)
WATER 3	-0.0951	(-0.5267 , 0.3366)	0.0020	(-0.1075 , 0.1115)

NOTE: IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

WATER 1 - OHIO RIVER WATER
WATER 2 - MUNICIPAL SECONDARY EFFLUENT
WATER 3 - ELECTROPLATING EFFLUENT

```
                 TABLE 62.  STUDY 39,SW 846 METHOD 3010,TRACE METALS,AA FLAME

                          EFFECT OF WATER TYPE ON CHROMIUM, CR ANALYSIS

                                    ** POINT ESTIMATES **

                        REAGENT WATER SLOPE:GAMMA(1) = 1.01956

     WATER                      INTERCEPT(WATER-REAGENT)     SLOPE(WATER-REAGENT)

               WATER 1                    0.0441                  -0.0242
               WATER 2                   -0.0321                   0.0232
               WATER 3                   -0.0052                   0.0017

                                   ** ANALYSIS OF VARIANCE **

                         SOURCE            DF   SUM OF SQUARES  MEAN SQUARE   F   PROB

                     REG(REAGENT)           1     235.77262      235.77262
                     REG(WATER/REAGENT)     6       0.07227        0.01205   1.07 0.3839
                     ERROR                144       1.62249        0.01127

                     TOTAL                151     237.46738



** TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **

                             INTERCEPT(WATER-REAGENT)              SLOPE(WATER-REAGENT)
     WATER                   ESTIMATE       INTERVAL          ESTIMATE        INTERVAL

               WATER 1         0.0441  ( -0.0396 ,   0.1279)   -0.0242  ( -0.0762 ,   0.0278)
               WATER 2        -0.0321  ( -0.1158 ,   0.0516)    0.0232  ( -0.0288 ,   0.0752)
               WATER 3        -0.0052  ( -0.0890 ,   0.0787)    0.0017  ( -0.0488 ,   0.0522)

NOTE:  IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN
       REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

       THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION
       AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

       WATER 1  -  OHIO RIVER WATER
       WATER 2  -  MUNICIPAL SECONDARY EFFLUENT
       WATER 3  -  ELECTROPLATING EFFLUENT
```

TABLE 63. STUDY 38, SW 846 METHOD 3010, TRACE METALS, AA FLAME

EFFECT OF WATER TYPE ON COBALT, CO ANALYSIS

** POINT ESTIMATES **

REAGENT WATER SLOPE:GAMMA(1) = 0.99892

WATER	INTERCEPT(WATER-REAGENT)	SLOPE(WATER-REAGENT)
WATER 1	-0.0534	0.0203
WATER 2	0.1202	-0.0557
WATER 3	-0.0349	0.0163

** ANALYSIS OF VARIANCE **

SOURCE	DF	SUM OF SQUARES	MEAN SQUARE	F	PROB
REG(REAGENT)	1	64.43237	64.43237		
REG(WATER/REAGENT)	6	0.07760	0.01293	4.52	0.0003
ERROR	146	0.41753	0.00286		
TOTAL	153	64.92750			

** TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **

WATER	INTERCEPT(WATER-REAGENT) ESTIMATE	INTERCEPT(WATER-REAGENT) INTERVAL	SLOPE(WATER-REAGENT) ESTIMATE	SLOPE(WATER-REAGENT) INTERVAL
WATER 1	-0.0534	(-0.1520 , 0.0451)	0.0203	(-0.0293 , 0.0699)
WATER 2	0.1202	(0.0196 , 0.2207)	-0.0557	(-0.1059 , -0.0055)
WATER 3	-0.0349	(-0.1381 , 0.0683)	0.0163	(-0.0340 , 0.0666)

NOTE: IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

WATER 1 - OHIO RIVER WATER
WATER 2 - MUNICIPAL SECONDARY EFFLUENT
WATER 3 - ELECTROPLATING EFFLUENT

TABLE 64. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

EFFECT OF WATER TYPE ON COPPER , CU ANALYSIS

** POINT ESTIMATES **

REAGENT WATER SLOPE:GAMMA(1) = 0.99355

WATER	INTERCEPT(WATER-REAGENT)	SLOPE(WATER-REAGENT)
WATER 1	0.0026	0.0025
WATER 2	0.0108	-0.0033
WATER 3	-0.0057	0.0041

** ANALYSIS OF VARIANCE **

SOURCE	DF	SUM OF SQUARES	MEAN SQUARE	F	PROB
REG(REAGENT)	1	255.45768	255.45768		
REG(WATER/REAGENT)	6	0.00621	0.00104	0.61	0.7252
ERROR	149	0.25455	0.00171		
TOTAL	156	255.71844			

** TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **

WATER	INTERCEPT(WATER-REAGENT) ESTIMATE	INTERCEPT(WATER-REAGENT) INTERVAL	SLOPE(WATER-REAGENT) ESTIMATE	SLOPE(WATER-REAGENT) INTERVAL
WATER 1	0.0026	(-0.0232 , 0.0284)	0.0025	(-0.0165 , 0.0215)
WATER 2	0.0108	(-0.0151 , 0.0367)	-0.0033	(-0.0228 , 0.0162)
WATER 3	-0.0057	(-0.0303 , 0.0190)	0.0041	(-0.0148 , 0.0231)

NOTE: IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

WATER 1 - OHIO RIVER WATER
WATER 2 - MUNICIPAL SECONDARY EFFLUENT
WATER 3 - ELECTROPLATING EFFLUENT

TABLE 65. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

EFFECT OF WATER TYPE ON IRON, FE ANALYSIS

```
                                  ** POINT ESTIMATES **

                    REAGENT WATER SLOPE:GAMMA(1) = 0.98995

WATER                  INTERCEPT(WATER-REAGENT)     SLOPE(WATER-REAGENT)

     WATER 1                  -0.3874                     0.2811
     WATER 2                  -0.1509                     0.1036
     WATER 3                  -0.2635                     0.2453


                               ** ANALYSIS OF VARIANCE **

                    SOURCE            DF   SUM OF SQUARES  MEAN SQUARE   F     PROB

               REG(REAGENT)            1     267.87445     267.87445
               REG(WATER/REAGENT)      6       4.10220       0.68370     8.95 0.0000
               ERROR                 158      12.06567       0.07637

               TOTAL                 165     284.04232


** TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **

   WATER                INTERCEPT(WATER-REAGENT)               SLOPE(WATER-REAGENT)
                        ESTIMATE        INTERVAL              ESTIMATE        INTERVAL

         WATER 1        -0.3874  ( -0.5626 ,  -0.2123)     0.2811  (  0.1339 ,   0.4283)
         WATER 2        -0.1509  ( -0.3255 ,   0.0238)     0.1036  ( -0.0457 ,   0.2530)
         WATER 3        -0.2635  ( -0.4344 ,  -0.0927)     0.2453  (  0.1020 ,   0.3886)

NOTE:  IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN
       REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

       THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION
       AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

       WATER 1  -  OHIO RIVER WATER
       WATER 2  -  MUNICIPAL SECONDARY EFFLUENT
       WATER 3  -  ELECTROPLATING EFFLUENT
```

```
TABLE 66.    STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

                       EFFECT OF WATER TYPE ON LEAD, PB ANALYSIS

                                  ** POINT ESTIMATES **

                         REAGENT WATER SLOPE:GAMMA(1) = 1.01012

  WATER               INTERCEPT(WATER-REAGENT)    SLOPE(WATER-REAGENT)

      WATER 1                  0.0654                   -0.0271
      WATER 2                 -0.0389                    0.0224
      WATER 3                  0.0184                   -0.0234

                              ** ANALYSIS OF VARIANCE **

                    SOURCE            DF   SUM OF SQUARES  MEAN SQUARE   F      PROB

              REG(REAGENT)             1     253.41402     253.41402
              REG(WATER/REAGENT)       6       0.13101       0.02197    4.80 0.0002
              ERROR                  154       0.70541       0.00458


              TOTAL                  161     254.25124



** TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **

                            INTERCEPT(WATER-REAGENT)              SLOPE(WATER-REAGENT)
  WATER                     ESTIMATE       INTERVAL             ESTIMATE       INTERVAL

      WATER 1                 0.0654   ( 0.0018 ,   0.1289)    -0.0271  ( -0.0589 ,   0.0047)
      WATER 2                -0.0389   ( -0.1040 ,  0.0262)     0.0224  ( -0.0097 ,   0.0545)
      WATER 3                 0.0184   ( -0.0467 ,  0.0834)    -0.0234  ( -0.0548 ,   0.0079)

NOTE:  IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN
       REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

       THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION
       AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

       WATER 1  -  OHIO RIVER WATER
       WATER 2  -  MUNICIPAL SECONDARY EFFLUENT
       WATER 3  -  ELECTROPLATING EFFLUENT
```

```
TABLE 67.  STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

                     EFFECT OF WATER TYPE ON MAGNESIUM, MG ANALYSIS

                                   ** POINT ESTIMATES **

           REAGENT WATER SLOPE:GAMMA(1) = 0.99861

   WATER                  INTERCEPT(WATER-REAGENT)      SLOPE(WATER-REAGENT)

       WATER 1                     -0.0484                    0.0107
       WATER 2                     -0.0821                    0.0210
       WATER 3                     -0.0311                    0.0044


                                ** ANALYSIS OF VARIANCE **

                  SOURCE             DF   SUM OF SQUARES  MEAN SQUARE   F    PROB

           REG(REAGENT)               1      91.12614      91.12614
           REG(WATER/REAGENT)         6       0.01717       0.00286   1.29 0.2631
           ERROR                    148       0.32707       0.00221


           TOTAL                    155      91.47038



 ** TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **

                              INTERCEPT(WATER-REAGENT)            SLOPE(WATER-REAGENT)
   WATER                      ESTIMATE        INTERVAL          ESTIMATE        INTERVAL

       WATER 1                 -0.0484  ( -0.1554 ,   0.0585)   0.0107  ( -0.0267 ,   0.0480)
       WATER 2                 -0.0821  ( -0.1892 ,   0.0249)   0.0210  ( -0.0163 ,   0.0584)
       WATER 3                 -0.0311  ( -0.1387 ,   0.0765)   0.0044  ( -0.0334 ,   0.0422)

NOTE:  IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN
       REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

       THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION
       AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

       WATER 1  -  OHIO RIVER WATER
       WATER 2  -  MUNICIPAL SECONDARY EFFLUENT
       WATER 3  -  ELECTROPLATING EFFLUENT
```

TABLE 68. STUDY 38, SW 846 METHOD 3010, TRACE METALS, AA FLAME

EFFECT OF WATER TYPE ON MANGANESE, MN ANALYSIS

```
                              ** POINT ESTIMATES **

                    REAGENT WATER SLOPE:GAMMA(1) = 0.98775

 WATER                        INTERCEPT(WATER-REAGENT)     SLOPE(WATER-REAGENT)

        WATER 1                        -0.0402                    0.0604
        WATER 2                        -0.0266                    0.0184
        WATER 3                         0.0025                    0.0022

                              ** ANALYSIS OF VARIANCE **

                  SOURCE            DF   SUM OF SQUARES  MEAN SQUARE   F    PROB

               REG(REAGENT)          1     283.86297     283.86297
               REG(WATER/REAGENT)    6       0.25102       0.04184    8.92 0.0000
               ERROR               148       0.69416       0.00469


               TOTAL               155     284.80815


  ** TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **

                               INTERCEPT(WATER-REAGENT)              SLOPE(WATER-REAGENT)
 WATER                         ESTIMATE       INTERVAL               ESTIMATE       INTERVAL

        WATER 1                -0.0402  ( -0.0837 ,   0.0032)    0.0604  (  0.0292 ,   0.0915)
        WATER 2                -0.0266  ( -0.0724 ,   0.0191)    0.0184  ( -0.0125 ,   0.0493)
        WATER 3                 0.0025  ( -0.0364 ,   0.0414)    0.0022  ( -0.0265 ,   0.0309)

NOTE:  IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN
       REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

       THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION
       AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

       WATER 1  -  OHIO RIVER WATER
       WATER 2  -  MUNICIPAL SECONDARY EFFLUENT
       WATER 3  -  ELECTROPLATING EFFLUENT
```

TABLE 69. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

EFFECT OF WATER TYPE ON MOLYBDENUM, NO ANALYSIS

```
                                   ** POINT ESTIMATES **

                         REAGENT WATER SLOPE:GAMMA(1) = 1.02218
     WATER                         INTERCEPT(WATER-REAGENT)     SLOPE(WATER-REAGENT)

              WATER 1                     0.0478                      -0.0259
              WATER 2                     0.1603                      -0.0531
              WATER 3                     0.0686                      -0.0298


                                  ** ANALYSIS OF VARIANCE **

                         SOURCE          DF   SUM OF SQUARES   MEAN SQUARE    F    PROB

                   REG(REAGENT)           1      330.25490      330.25490
                   REG(WATER/REAGENT)     6        0.18093        0.03149   2.85 0.0117
                   ERROR                147        1.62135        0.01103


                   TOTAL                154      332.06517



  ** TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **

                                   INTERCEPT(WATER-REAGENT)             SLOPE(WATER-REAGENT)
     WATER                          ESTIMATE      INTERVAL            ESTIMATE       INTERVAL

              WATER 1                0.0478  ( -0.0629 ,   0.1586)   -0.0259  ( -0.0697 ,   0.0178)
              WATER 2                0.1603  (  0.0497 ,   0.2709)   -0.0531  ( -0.0968 ,  -0.0093)
              WATER 3                0.0686  ( -0.0377 ,   0.1748)   -0.0298  ( -0.0716 ,   0.0119)

NOTE:  IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN
       REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

       THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION
       AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.
       WATER 1  -  OHIO RIVER WATER
       WATER 2  -  MUNICIPAL SECONDARY EFFLUENT
       WATER 3  -  ELECTROPLATING EFFLUENT
```

TABLE 70. STUDY 38, SW 846 METHOD 3010, TRACE METALS, AA FLAME

EFFECT OF WATER TYPE ON NICKEL, NI ANALYSIS

```
                              ** POINT ESTIMATES **

            REAGENT WATER SLOPE:GAMMA(1) = 1.01510

  WATER               INTERCEPT(WATER-REAGENT)     SLOPE(WATER-REAGENT)

        WATER 1                -0.0034                    -0.0045
        WATER 2                -0.0011                     0.0126
        WATER 3                 0.0151                    -0.0031


                           ** ANALYSIS OF VARIANCE **

              SOURCE            DF   SUM OF SQUARES  MEAN SQUARE   F     PROB

            REG(REAGENT)          1     218.34402     218.34402
            REG(WATER/REAGENT)    6       0.01736       0.00289    0.43 0.8562
            ERROR               172       1.14968       0.00668

            TOTAL               179 .   219.51107


** TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **

                               INTERCEPT(WATER-REAGENT)           SLOPE(WATER-REAGENT)
  WATER                        ESTIMATE       INTERVAL          ESTIMATE       INTERVAL

        WATER 1                -0.0034  ( -0.0480 ,   0.0412)  -0.0045  ( -0.0447 ,   0.0356)
        WATER 2                -0.0011  ( -0.0490 ,   0.0468)   0.0126  ( -0.0293 ,   0.0545)
        WATER 3                 0.0151  ( -0.0328 ,   0.0630)  -0.0031  ( -0.0450 ,   0.0380)
```

NOTE: IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER (INTERCEPT/SLOPE).

THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

WATER 1 - OHIO RIVER WATER
WATER 2 - MUNICIPAL SECONDARY EFFLUENT
WATER 3 - ELECTROPLATING EFFLUENT

```
TABLE 71.  STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

           EFFECT OF WATER TYPE ON POTASSIUM, K ANALYSIS

                        ** POINT ESTIMATES **

           REAGENT WATER SLOPE:GAMMA(1) = 0.99110

WATER                 INTERCEPT(WATER-REAGENT)    SLOPE(WATER-REAGENT)

      WATER 1                -0.0389                      0.0059
      WATER 2                 0.0981                     -0.0227
      WATER 3                -0.0227                      0.0171


                       ** ANALYSIS OF VARIANCE **

                SOURCE            DF  SUM OF SQUARES  MEAN SQUARE   F    PROB

           REG(REAGENT)            1     72.88677       72.88677
           REG(WATER/REAGENT)      6      0.09795        0.01633    1.25 0.2820
           ERROR                 152      1.97833        0.01302


           TOTAL                 159     74.96305



** TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **

                         INTERCEPT(WATER-REAGENT)             SLOPE(WATER-REAGENT)
WATER                    ESTIMATE       INTERVAL            ESTIMATE        INTERVAL

      WATER 1            -0.0389  ( -0.2975 ,   0.2198)   0.0059  ( -0.0931 ,   0.1049)
      WATER 2             0.0981  ( -0.1623 ,   0.3585)  -0.0227  ( -0.1218 ,   0.0765)
      WATER 3            -0.0227  ( -0.2799 ,   0.2345)   0.0171  ( -0.0812 ,   0.1154)

NOTE:  IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN
       REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

       THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION
       AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

       WATER 1  -  OHIO RIVER WATER
       WATER 2  -  MUNICIPAL SECONDARY EFFLUENT
       WATER 3  -  ELECTROPLATING EFFLUENT
```

```
TABLE 72.  STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

                    EFFECT OF WATER TYPE ON SILVER, AG ANALYSIS

                                   ** POINT ESTIMATES **

                REAGENT WATER SLOPE:GAMMA(1) = 0.57832

WATER                  INTERCEPT(WATER-REAGENT)     SLOPE(WATER-REAGENT)

                                -0.4767                    -0.0876
        WATER 1                 -0.1253                     0.0204
        WATER 2                 -1.4421                    -0.3991
        WATER 3


                              ** ANALYSIS OF VARIANCE **

                SOURCE            DF   SUM OF SQUARES  MEAN SQUARE    F     PROB

            REG(REAGENT)           1      93.41512      93.41512
            REG(WATER/REAGENT)     6      39.23251       6.53875     21.97 0.0000
            ERROR                163      48.51320       0.29763


            TOTAL                170     181.16082



** TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **
                            INTERCEPT(WATER-REAGENT)              SLOPE(WATER-REAGENT)
                            ESTIMATE       INTERVAL              ESTIMATE      INTERVAL
WATER
                            -0.4767  ( -0.7856 ,  -0.1678)   -0.0876  ( -0.2979 ,   0.1228)
        WATER 1             -0.1253  ( -0.4067 ,   0.1561)    0.0204  ( -0.1778 ,   0.2186)
        WATER 2             -1.4421  ( -1.7972 ,  -1.0870)   -0.3991  ( -0.6323 ,  -0.1660)
        WATER 3

NOTE:  IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN
       REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

       THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION
       AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

       WATER 1  -  OHIO RIVER WATER
       WATER 2  -  MUNICIPAL SECONDARY EFFLUENT
       WATER 3  -  ELECTROPLATING EFFLUENT
```

TABLE 73. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

EFFECT OF WATER TYPE ON SODIUM, NA ANALYSIS

** POINT ESTIMATES **

REAGENT WATER SLOPE:GAMMA(1) = 1.00037

WATER	INTERCEPT(WATER-REAGENT)	SLOPE(WATER-REAGENT)
WATER 1	0.0568	-0.0127
WATER 2	-0.0810	0.0084
WATER 3	-0.4418	0.0629

** ANALYSIS OF VARIANCE **

SOURCE	DF	SUM OF SQUARES	MEAN SQUARE	F	PROB
RES(REAGENT)	1	73.91471	73.91471		
REG(WATER/REAGENT)	6	0.22479	0.03747	7.76	0.0000
ERROR	142	0.68571	0.00483		
TOTAL	149	74.82521			

** TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **

WATER	INTERCEPT(WATER-REAGENT)		SLOPE(WATER-REAGENT)	
	ESTIMATE	INTERVAL	ESTIMATE	INTERVAL
WATER 1	0.0568	(-0.2822 , 0.3957)	-0.0127	(-0.0736 , 0.0483)
WATER 2	-0.0810	(-0.3991 , 0.2371)	0.0084	(-0.0489 , 0.0656)
WATER 3	-0.4418	(-0.7787 , -0.1050)	0.0629	(0.0025 , 0.1234)

NOTE: IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

WATER 1 - OHIO RIVER WATER
WATER 2 - MUNICIPAL SECONDARY EFFLUENT
WATER 3 - ELECTROPLATING EFFLUENT

TABLE 74. STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

EFFECT OF WATER TYPE ON THALLIUM, TI ANALYSIS

** POINT ESTIMATES **

REAGENT WATER SLOPE:GAMMA(1) = 1.00883

WATER	INTERCEPT(WATER-REAGENT)	SLOPE(WATER-REAGENT)
WATER 1	-0.0248	0.0047
WATER 2	-0.0120	0.0016
WATER 3	0.0150	-0.0067

** ANALYSIS OF VARIANCE **

SOURCE	DF	SUM OF SQUARES	MEAN SQUARE	F	PROB
REG(REAGENT)	1	288.11314	288.11314		
REG(WATER/REAGENT)	6	0.01748	0.00291	1.56	0.1603
ERROR	185	0.34487	0.00186		
TOTAL	192	288.47549			

** TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **

WATER	INTERCEPT(WATER-REAGENT) ESTIMATE	INTERCEPT(WATER-REAGENT) INTERVAL	SLOPE(WATER-REAGENT) ESTIMATE	SLOPE(WATER-REAGENT) INTERVAL
WATER 1	-0.0248	(-0.0666 , 0.0170)	0.0047	(-0.0151 , 0.0245)
WATER 2	-0.0120	(-0.0530 , 0.0290)	0.0016	(-0.0178 , 0.0210)
WATER 3	0.0150	(-0.0255 , 0.0555)	-0.0067	(-0.0259 , 0.0126)

NOTES: IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

WATER 1 - OHIO RIVER WATER
WATER 2 - MUNICIPAL SECONDARY EFFLUENT
WATER 3 - ELECTROPLATING EFFLUENT

```
                TABLE 75.  STUDY 38,SW 846 METHOD 3010,TRACE METALS,AA FLAME

                        EFFECT OF WATER TYPE ON VANADIUM, V ANALYSIS

                                ** POINT ESTIMATES **

           REAGENT WATER SLOPE:GAMMA(1) = 0.99636

                      INTERCEPT(WATER-REAGENT)    SLOPE(WATER-REAGENT)

   WATER 1                    0.0323                    -0.0036
   WATER 2                   -0.0324                     0.0071
   WATER 3                   -0.0067                     0.0044


                              ** ANALYSIS OF VARIANCE **

           SOURCE            DF   SUM OF SQUARES  MEAN SQUARE    F     PROB

         REG(REAGENT)          1      365.81226     365.81226
         REG(WATER/REAGENT)    6        0.02417       0.00403     0.67 0.6756
         ERROR               140        0.84433       0.00603


         TOTAL               147      366.68077



 ** 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **

                        INTERCEPT(WATER-REAGENT)              SLOPE(WATER-REAGENT)
                        ESTIMATE        INTERVAL            ESTIMATE       INTERVAL

   WATER 1               0.0323  ( -0.0717 ,    0.1363)   -0.0036  ( -0.0337 ,    0.0265)
   WATER 2              -0.0324  ( -0.1314 ,    0.0665)    0.0071  ( -0.0217 ,    0.0359)
   WATER 3              -0.0067  ( -0.1088 ,    0.0954)    0.0044  ( -0.0255 ,    0.0343)

NOTE:  IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN
       REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

       THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION
       AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

       WATER 1     OHIO RIVER WATER
       WATER 2     MUNICIPAL SECONDARY EFFLUENT
       WATER 3     ELECTROPLATING EFFLUENT
```

```
TABLE 76.   STUDY 39,SW 846 METHOD 3010,TRACE METALS,AA FLAME

                        EFFECT OF WATER TYPE ON ZINC, ZN ANALYSIS

                                   ** POINT ESTIMATES **

                  REAGENT WATER SLOPE:GAMMA(1) = 0.97879

WATER                       INTERCEPT(WATER-REAGENT)     SLOPE(WATER-REAGENT)

          WATER 1                      0.1190                    0.4964
          WATER 2                      0.0144                   -0.0291
          WATER 3                     -0.0203                    0.0103


                               ** ANALYSIS OF VARIANCE **

                  SOURCE            DF   SUM OF SQUARES  MEAN SQUARE   F      PROB

              REG(REAGENT)           1     117.38378      117.38378
              REG(WATER/REAGENT)     6       7.43511        1.23918    19.82 0.0000
              ERROR                129       8.06559        0.06252


              TOTAL                136     132.88448



TABLE OF 95% CONFIDENCE INTERVALS FOR THE DIFFERENCES BETWEEN INTERCEPTS AND THE DIFFERENCES BETWEEN SLOPES **

                            INTERCEPT(WATER-REAGENT)             SLOPE(WATER-REAGENT)
WATER                        ESTIMATE        INTERVAL           ESTIMATE        INTERVAL

          WATER 1             0.1190  ( -0.1644 ,    0.4024)    0.4964  (  0.2928 ,    0.7000)
          WATER 2             0.0144  ( -0.2538 ,    0.2825)   -0.0291  ( -0.2241 ,    0.1658)
          WATER 3            -0.0203  ( -0.2655 ,    0.2249)    0.0103  ( -0.1732 ,    0.1938)

NOTE:  IF ZERO IS CONTAINED WITHIN A GIVEN CONFIDENCE INTERVAL THEN THERE IS NO STATISTICAL SIGNIFICANCE BETWEEN
       REAGENT WATER AND THE CORRESPONDING WATER FOR THE ASSOCIATED PARAMETER(INTERCEPT/SLOPE).

       THE SLOPE AND INTERCEPT ESTIMATES FROM THIS ANALYSIS ARE NOT THE SAME AS THOSE OBTAINED FROM THE PRECISION
       AND MEAN RECOVERY REGRESSIONS PERFORMED EARLIER.

       WATER 1  -  OHIO RIVER WATER
       WATER 2  -  MUNICIPAL SECONDARY EFFLUENT
       WATER 3  -  ELECTROPLATING EFFLUENT
```

APPENDIX E

METHOD DETECTION LIMIT (MDL)
RAW DATA

TABLE 77. MEASURED MDLs IN REAGENT WATER BY LABORATORY (ug/mL)

Element	Laboratory Number 1	2	3	4	5	6	7	8	9	Avg
Al	0	0.3	0.326	1.79*	0.187	0.25	0.151	0.62	0.262	0.299
Ba	0	0.08	0.75*	0.103	0.071	0.01	0.104	0.32	0.308	0.142
Be	0.010	0.007	0.013	0.008	0.0018	0.016	0.0054	0	0.011	0.009
Ca	0.028	0.02	0.046	0.029	0.0311	0.011	0.0189	0.040	0.038	0.029
Fe	0.046	0.02	0.041	0.16	0.0208	0.11	0.0509	0.064	0.030	0.060
K	0.0036	0.0004	0.003	0.002	0.0009	0.002	0.0015	0.003	0.002	0.0020
Mg	0.0069	0.0008	0.007	0.005	0.0022	0.002	0.0018	0.004	0.002	0.0035
Mo	0	0.005	0.103	0.28	0.0949	0.044	0.0673	0.34	0.261	0.149
Na	0.0067	0.007	0	0.042	0.001	0.003	0.0039	0.0054	0.005	0.015
Sb	0.18	0.2	0	0.72	0.139	0.25	0.168	0.55	0.364	0.378
V	0	0	0	0.64	0.355	0.16	0.254	0.37	0	0.356
Ag	0.22*	0.009	0.051	0.020	0.016	0.031	0.0147	0	0.014	0.022
Cd	0.009	0.005	0	0.034	0.0056	0.027	0.0267	0.016	0.009	0.016
Co	0.042	0.03	0.531*	0.098	0.0318	0.11	0.0301	0.10	0.034	0.060
Cr	0.055	0.05	0.154	0.057	0.0457	0.15	0	0.16	0.095	0.096
Cu	0.024	0.02	0.018	0.008	0.0123	0.023	0	0	0.019	0.018
Mn	0.028	0.06	0.013	0.012	0.0141	0.015	0.0126	0.029	0.015	0.022
Ni	0.043	0.02	0.027	0.067	0.0159	0.23*	0.0712	0.074	0.046	0.046
Pb	0.10	0.02	0.349	0.36	0.0439	0.24	0.456	0	0.345	0.239
Tl	0	0.1	0.136	0.10	0.0487	0.19	0	0.807*	0.106	0.114
Zn	0.0099	0.005	0	0.02	0.0022	0.011	0.0119	0.008	0.023	0.011

* Outliers by Thompson's "T" test.

TABLE 78. MEASURED MDLs IN OHIO RIVER WATER BY LABORATORY (ug/mL)

	Laboratory Number									
Element	1	2	3	4	5	6	7	8	9	Avg
Al	0	0.4	0.313	1.86*	0.194	0.22	0.141	0.63	0.255	0.256
Ba	0	0.08	0.60	0.096	0.077	0.01	0.105	0.33	0.300	0.200
Be	0.011	0.007	0.014	0.008	0.0037	0.027	0.0054	0	0.010	0.011
Ca	--	--	--	--	--	--	--	--	--	--
Fe	--	--	--	--	--	--	--	--	--	--
K	--	--	--	--	--	--	--	--	--	--
Mg	--	--	--	--	--	--	--	--	--	--
Mo	0	0.05	0.197	0.31	0.119	0.044	0.0694	0.33	0.401	0.268
Na	--	--	--	--	--	--	--	--	--	--
Sb	0.18	0.2	0	0.73	0.128	0.27	0.645	0.59	0.353	0.350
V	0	0	0	0.52	0.353	0.16	0.258	0.37	0	0.332
Ag	0.024	0.009	0.051	0.007	0.0139	0.031	0.0158	0	0.014	0.020
Cd	0.0091	0.005	0	0.035	0.005	0.028	0.0264	0.016	0.009	0.017
Co	--	--	--	--	--	--	--	--	--	--
Cr	0.051	0.04	0.175	0.028	0.040	0.15	0	0.15	0.108	0.093
Cu	0.023	0.02	0.019	0.008	0.0142	0.023	0	0	0.019	0.018
Mn	0.025	0.06*	0.014	0.012	0.0144	0.017	0.0121	0.028	0.015	0.017
Ni	0.044	0.02	0.028	0.072	0.016	0.24*	0.072	0.073	0.048	0.047
Pb	0.10	0.2	0.341	0.25	0.089	0.24	0.459	0	0.307	0.248
Tl	0	0.1	0.136	0.11	0.052	0.18	0	0.72*	0.106	0.114
Zn	--	--	--	--	--	--	--	--	--	--

* Outliers by Thompson's "T" test.

-- No MDL determined because of high background concentration.

TABLE 79. MEASURED MDLs IN MUNICIPAL SECONDARY EFFLUENT BY LABORATORY (ug/mL)

Element	Laboratory Number 1	2	3	4	5	6	7	8	9	Avg
Al	0	0.3	0.341	1.73*	0.18	0.21	0.137	0.61	0.248	0.247
Ba	0	0.08	0.50	0.094	0.057	0.010	0.103	0.36	0.268	0.184
Be	0.010	0.007	0.014	0.009	0.0035	0.019	0.0055	0	0.011	0.010
Ca	--	--	--	--	--	--	--	--	--	--
Fe	--	--	--	--	--	--	--	--	--	--
K	--	--	--	--	--	--	--	--	--	--
Mg	--	--	--	--	--	--	--	--	--	--
Mo	0	0.05	0.179	0.30	0.095	0.044	0.070	0.31	0.280	0.116
Na	--	--	--	--	--	--	--	--	--	--
Sb	0.18	0.2	0	0.72	0.144	0.27	0.590	0.57	0.369	0.380
V	0	0	0	0.60	0.399	0.16	0.259	0.35	0	0.354
Ag	0.023	0.009	0.051	0.007	0.0103	0.030	0.0156	0	0.014	0.020
Cd	0.0086	0.005	0	0.035	0.0054	0.028	0.0272	0.016	0.009	0.017
Co	--	--	--	--	--	--	--	--	--	--
Cr	--	--	--	--	--	--	--	--	--	--
Cu	0.023	0.02	0.018	0.008	0.0146	0.023	0	0	0.019	0.018
Mn	0.024	0.06	0.014	0.01	0.0112	0.016	0.0128	0.028	0.016	0.017
Ni	--	--	--	--	--	--	--	--	--	--
Pb	0.095	0.2	0.366	0.32	0.0958	0.24	0.451	0	0.285	0.257
Tl	0	0.1	0.14	0.11	0.0727	0.16	0	0.74*	0.101	0.114
Zn	--	--	--	--	--	--	--	--	--	--

* Outliers by Thompson's "T" test.

-- No MDL determined because of high background concentration.

TABLE 80. MEASURED MDLs IN ELECTROPLATING EFFLUENT BY LABORATORY (ug/mL)

Element	Laboratory Number 1	2	3	4	5	6	7	8	9	Avg
Al	0	0.34	0.375	2.02*	0.188	0.21	0.133	0.65	0.255	0.307
Ba	0	0.08	0.479	0.094	0.071	0.010	0.104	0.38	0.34	0.195
Be	0.0099	0.007	0.014	0.008	0.0035	0.018	0.0054	0	0.010	0.010
Ca	--	--	--	--	--	--	--	--	--	--
Fe	--	--	--	--	--	--	--	--	--	--
K	--	--	--	--	--	--	--	--	--	--
Mg	--	--	--	--	--	--	--	--	--	--
Mo	0	0.05	0.156	0.27	0.0967	0.044	0.0683	0.31	0.238	0.153
Na	--	--	--	--	--	--	--	--	--	--
Sb	0.18	0.2	0	0.74	0.120	0.27	0.629	0.58	0.353	0.384
V	0	0	0	0.75	0.554	0.16	0.253	0.36	0	0.415
Ag	0.020	0.009	0.047	0.008	0.0162	0.030	0.0171	0	0.020	0.021
Cd	0.0088	0.005	0	0.036	0.0058	0.028	0.0282	0.016	0.009	0.017
Co	--	0.03	0.559*	0.094	0.0280	0.12	0.0294	0.10	0.042	0.063
Cr	--	0.04	0.158	0.028	0.0467	0.13	0	0.14	0.099	0.092
Cu	0.023	0.02	0.021	0.008	0.144*	0.023	0	0	0.018	0.019
Mn	0.025	0.06*	0.014	0.011	0.014	0.017	0.0129	0.030	0.016	0.018
Ni	--	0.02	0.029	0.070	0.016	0.23*	0.0812	0.073	0.050	0.049
Pb	0.098	0.2	0.366	0.31	0.0998	0.24	0.461	0	0.325	0.263
Tl	0	0.1	0.146	0.10	0.0715	0.19	0	0.72*	0.102	0.118
Zn	--	--	--	--	--	--	--	--	--	--

* Outliers by Thompson's "T" test.

-- No MDLs determined because of high background concentration.